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Modulus
Tan delta

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The objective of this work is to automate data taking and data reduction on a Rheovibron Dynamic Viscoelastometer. A computer program with the necessary hardware has been developed to control tension on the sample, to monitor power to heaters, to control a temperature scan, and to acquire data on polymeric solids from which temperature dependence of a dynamic modulus and tangent of the loss angle (tan δ) can be calculated.

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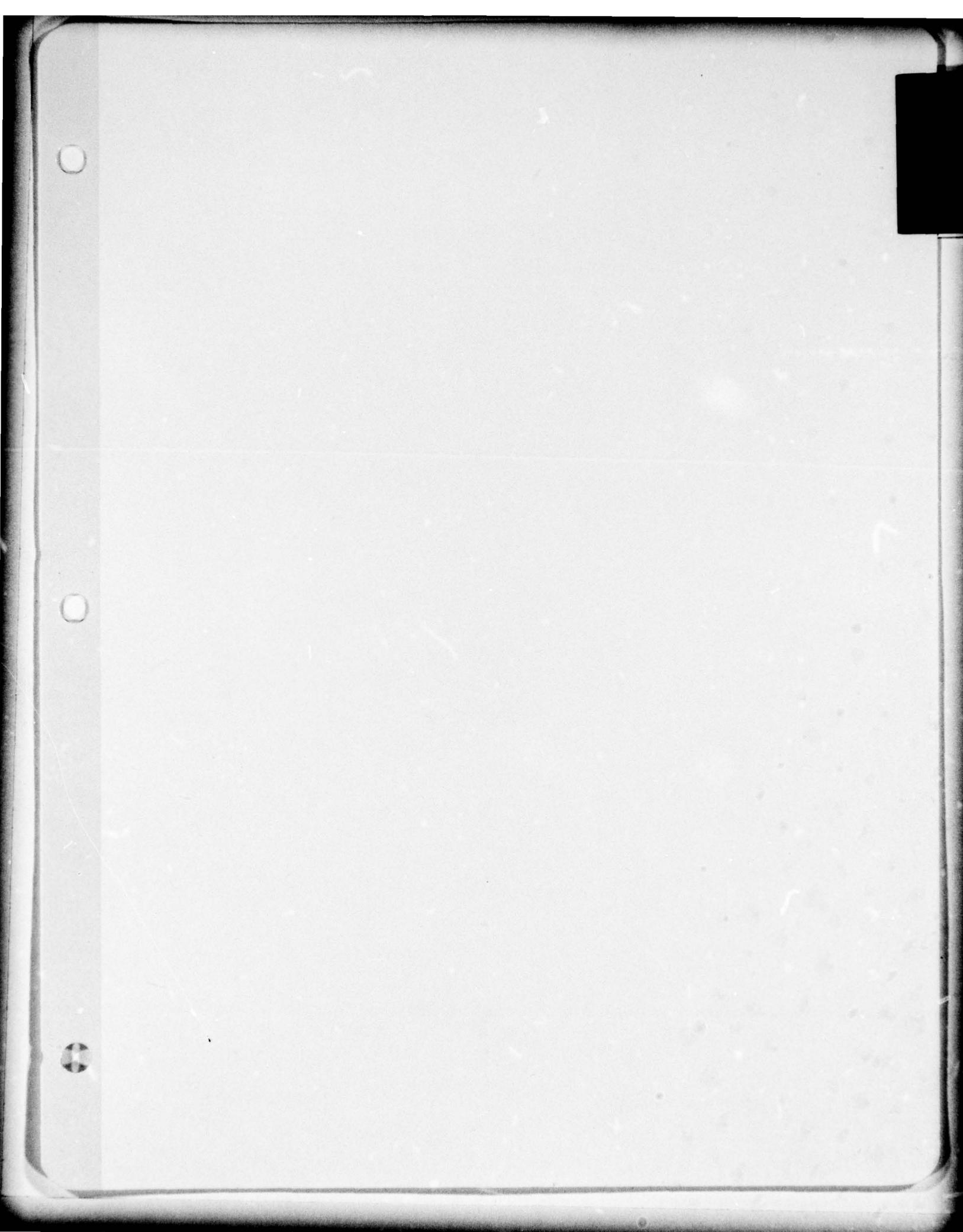
Report N00014-77-C-0421 #1-78

Development of Electronics, Mechanical Fixtures and Computer Software for Automation of Dynamic Mechanical Measurements on Polymer Solids and Computer Reduction of Data

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28 August 78   73 p.

Final Technical Report, - Final

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Office of Naval Research
Arlington, Virginia

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INTRODUCTION

Dynamic mechanical measurements on polymers and other viscoelastic materials are an attractive means of measuring fundamental relaxations which reflect molecular motions in the sample. Such measurements are useful in studying effects of many parameters such as molecular weight, crystallinity, crosslinking, additives and plasticizers, copolymerization and blending on mechanical performance.

One of the most popular commercial instruments for making dynamic measurements is the Rheovibron which was designed and developed by Professor Motowo Takayanagi of Kyushu University, Fukuoka, Japan. His publications involving the instrument have given him recognition, worldwide, as a leading polymer scientist. The instrument was built and offered for sale commercially by Toyo Baldwin Co. Ltd. (formerly Toyo Measuring Instruments Co. Ltd.) of Tokyo, Japan.

The most serious obstacles to the use of the Rheovibron are its labor intensity (a single scan on one sample can require as much as 5 hours of undivided operator attention) and the high level of operator skill required to obtain reproduceable measurements.

This development was undertaken to automate the instrument both with regard to data acquisition and to data reduction. By employing a dedicated computer, we planned to improve both the accuracy and precision of measurement by eliminating human errors which are most prevalent in applying tension to the sample, and by eliminating operator decisions and incorporating such decisions in the computer program.

The instrument is designed to impose a sinusoidal strain, in tension, on the sample and to measure the sinusoidal stress response of the material with respect to amplitude and phase. From these data one can determine a complex modulus (E*) at known frequency and temperature and a damping factor which is defined as the ratio of the quadrature and in-phase components of the complex modulus and which also represents the tangent of the phase angle which exists between the sinusoidal deformation and stress response of the sample.
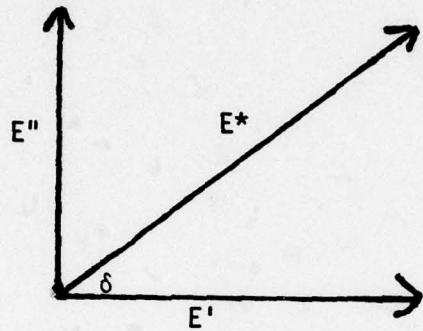
$$E^* = \frac{\text{Dynamic Stress Amp.}}{\text{Dynamic Strain Amp.}} \qquad \text{Tan } \delta \quad \frac{E''}{E'}$$

Figure 1.

The amplitude of the sinusoidal deformation is made small (1.0×10^{-3} cm, approximately .03 - 0.05% strain) to insure linear viscoelastic response The frequency is maintained constant for each data point.

Data are usually taken over a range of temperature and sometimes over a limited range of frequencies. A typical range of temperature is -150°C to as far above the glass transition temperature as the operator's skill can achieve. The limit is usually determined by sag and creep of the sample under gravity and tension making further data taking impossible. The normal frequencies of the instrument are 3.5, 11, 35, 110 Hz.

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OBJECTIVES OF THIS WORK

GENERAL

1. Reduce labor intensity of measurement by automating control functions and data taking.

2. Improve the precision of measurement especially with respect to tan δ.

3. Extend the range of tan δ measurements.

4. Provide a computer program for data reduction.

DESCRIPTION OF THE AUTOMATION SYSTEM

The system retains and uses the standard electronics associated with manual operation of the Rheovibron. Transducer excitation, electromagnetic driver excitation, and reference signal are all furnished to the system by the standard instrument. The automatic control functions are sample tension and temperature. Data taking and data reduction are also automatically handled by the system.

The automation system is designed around a computer program which is implemented with a Hewlett-Packard 9825A Calculator, interfaced for control and data acquisition to a Hewlett-Packard C940B Multiprogrammer. Commands in the program instruct the instrument through the multiprogrammer to initiate controls over the experiment.

Other program commands instruct the miltiprogrammer to acquire data and return it to the calulator for manipulation and/or storage. A separate program is provided for data reduction and a line printer and/or plotter can be provided as optional equipment.

The standard phase angle measuring system has been replaced with a lock-in amplifier (Princeton Applied Research Model 2504) to simplify automation of the measurement, to improve resolution of small angles, and to extend the range of tan δ measurements.

The simplified system is shown, schematically, in Figure 2. A Rheovibron interface, on command from the calculator, uses a signal from either the load or displacement transducer to the lock-in amplifier where it is measured. The measured result is passed through the Amp, Phase, Temp. and Relay cards to the Voltage Monitor where it is digitized and stored in the calculator memory. The two signals are alternately measured throughout the test run. The interface also passes the load transducer signal through the tension amplifier and relay card to the voltage monitor for digitizing and to the calculator

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where it is compared for tension limits. If the tension requires correction, a signal from the calculator through the relay card commands the motor to increase or decrease sample tension the proper number of steps. Temperature at the sample is detected by a platinum resistance thermometer. The signal is read by the program through the temperature translator, the Amp, Phase, Temp card, the Relay card, and voltage monitor to the calculator. The measured temperature is compared to values in the calculator program which control power to the environmental chamber heaters. Power is supplied at levels sufficient to maintain temperature increase of about 1°C/min.

BLOCK DIAGRAM OF SYSTEM

Figure 2

4
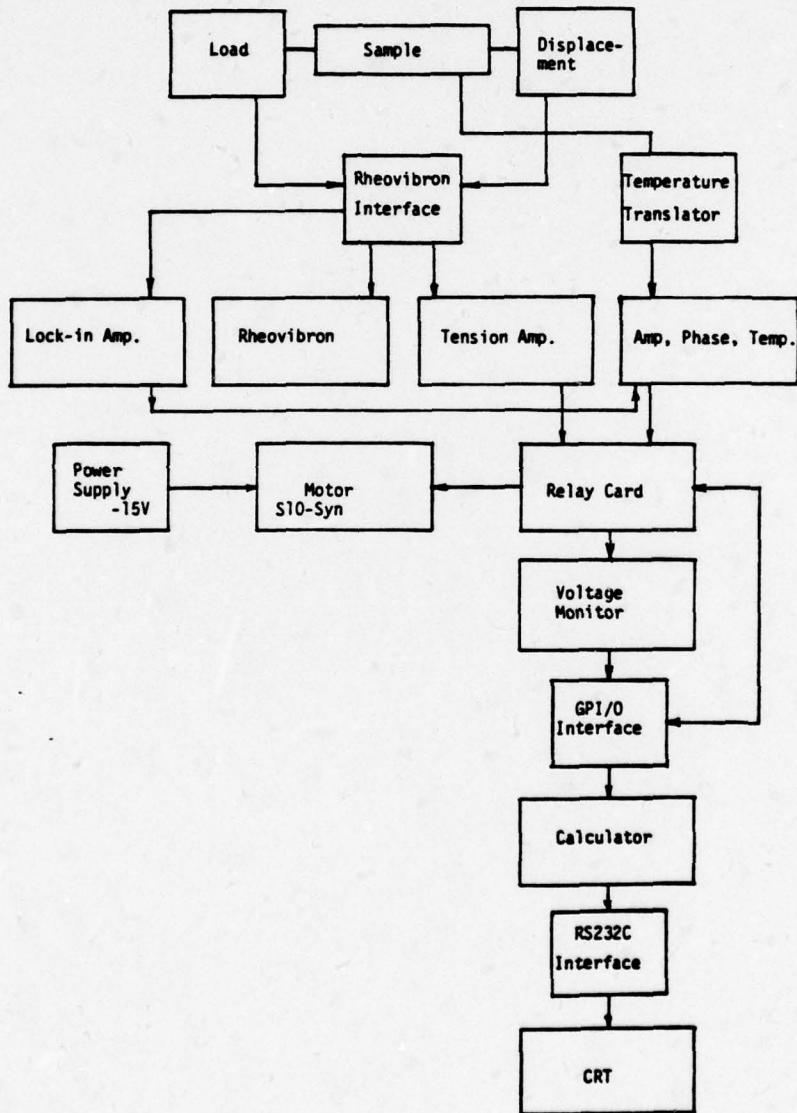
The Tension Amplifier, Amp, Phase, Temp. card, Relay card, Motor Power Supply and Amplifier, and Voltage Monitor are all contained in the Multiprogrammer.

The following data values are measured and recorded on a magnetic tape cassette for eventual data reduction:

Strain phase angle, degrees

Strain amplitude, cm.

Sample length, cm.

Average static tension, dynes

Temperature °C

Stress phase angle, degrees

Stress amplitude, dynes

MEASUREMENT FEATURES

1. Temperature

Temperature is detected by a 100-ohm platinum resistance thermometer (RDF #80RB) and is measured and transmitted through the multiprogrammer to the calculator by a temperature transmitter card (RDF Model 2050). This card provides a linearied output signal of 1 mv per degree. Accuracy is ±1°C over a full scale range (-180°C to +650°C).

The temperature chamber is designed with sufficient thermal mass to make control unnecessary from -150°C to approximately -60°C. Natural heat absorption after removal of liquid N_2 cooling causes the system to increase in temperature at a rate very close to 1°/min. which is about ideal for data acquisition. Above -30°C some power must be supplied to the heaters to maintain the rate of temperature rise. This is handled by a heater control system consisting of two solid state relays which proportion power to the heaters in amounts depending on needs. Power requirements are divided into four approximate levels:

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5%, 30%, 50% and 70%. Each level covers a specific range of temperature and the program automatically switches to the next higher power when each temperature level is reached. The system maintains a rate of temperature increase of approximately 1°C/min.

2. Modulus and Phase Measurement

A lock-in amplifier (Princeton Applied Research Model 5204) is used to measure in-phase and quadrature amplitudes of both the load and displacement signals and the phase relation of each signal with respect to a reference from the Rheovibron. The calculator program sends a command to the multiprogrammer to switch on the load signal to the lock-in amplifier which, after a program delay for settling, reads the in-phase and quadrature components with respect to the reference, and stores them in the calculator memory.
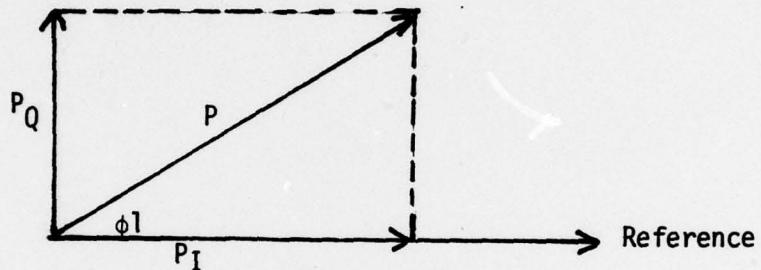
Figure 3

After these values are stored, the multiprogrammer receives a command to switch the dynamic displacement signal into the lock-in amplifier where it repeats the same measurements on these signals and stores the results in the calculator:

Figure 4

6
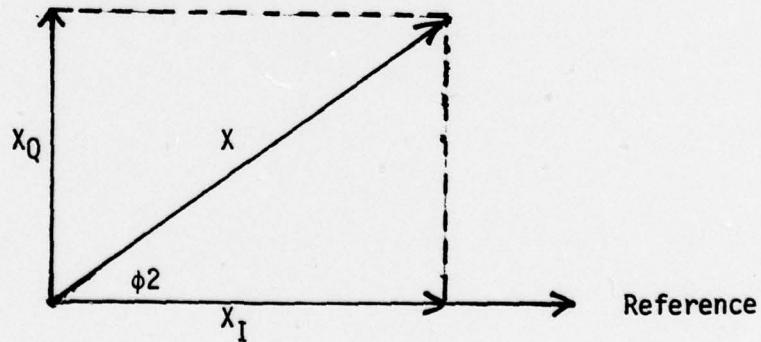
The calculator program than computes the voltage magnitudes of both the load and displacement signals, P and X, and the phase angle, δ, between the load and displacement.

$$P = \sqrt{P_I{}^2 + P_Q{}^2}$$

$$X = \sqrt{X_I{}^2 + X_Q{}^2}$$

$$\delta = \phi 1 - \phi 2 = \tan^{-1} \frac{P_Q}{P_I} - \tan^{-1} \frac{X_Q}{X_I}$$

The load and displacement signals are scaled by the calibration factors stored in the program and the complex modulus, E, is computed and displayed on the calculator. At the same time the tangent of the loss angle, tan δ, is displayed with the temperature. This modulus is uncorrected for instrument compliance but is corrected for changes in sample length which will be discussed in the section on tensioning the sample.

3. Sample Tension Control

The type of sample most frequently used (thin films, fibers, monofilaments, etc.) on the Rheovibron will not support a compressive load without buckling. This condition would, of course, distort the negative portion of the sine wave making measurements impossible. To alleviate this situation the sample must be maintained under sufficient tension to eliminate buckling but insufficient to introduce non-linearity or excessive creep into the experiment. The amount of tension is critical to the measurement.

The tension control system uses a stepping motor (Superior Electric Slo-Syn Model 5850-1008) to add or subtract small increments of tension to the sample. The motor receives its commands from the calculator. Each step of the motor represents a change in length of the sample of 1.25×10^{-4} cm. The program computes the modulus of the sample and uses this value to establish maximum and minimum limits of tension. It then calculates the magnitude of a single step to determine its load value and computes the number of steps required to put the sample tension within limits. The program then instructs the motor, through the multiprogrammer, to initiate the calculated number of steps, either + or -. The tension is measured and corrected twice during the taking of each datum point. As the sample softens, the program changes tension limits. An up and down count is stored in a register which, when multiplied by the value of each step, is added to, or subtracted from the initial sample length.

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TYPICAL TEST PROCEDURE

Following is a step by step description of a typical test.

1. Turn on power to all systems and allow at least a 20 minute warmup.

2. Prepare the sample using care that its size does not exceed the instrument force capability as explained on pages 41-44 of Rheovibron Manual #68. Thickness and width, in cm, are carefully measured and recorded.

3. Install sample in the grips using extreme care that it is straight and will not develop side thrusts or distortions when tension is applied. This is very important and has great bearing on quality of the data.

4. Load the control program into the calculator and strike RUN.

5. Type in the parameters requested by the program. After each entry, strike CONTINUE.

6. At the end of the parameters routine the calculator will display "Ready for Cooldown?". If yes, strike CONTINUE.

7. Add liquid nitrogen to the chamber and to the Dewar cooling the N_2 gas. Start gas flow at high rate, about 16-18 SCFH on the flowmeter. Keep a good supply of liquid N_2 in both the chamber container and the Dewar during cooldown.

8. The program will follow the course of cooldown, automatically adjusting tension as the sample shrinks. When starting temperature is reached, the calculator will display ...Minimum Temperature..., and the program will halt.

9. Reduce the nitrogen gas flow to about 6-8 SCFH and allow the liquid N_2 to boil off the temperature chamber tank. The main data taking program is initiated by striking CONTINUE.

10. From this point data taking and tension control are automatic. The temperature will rise approximately 1°C/min. from the heat obtained from surrounding. At about -60°C, remove the copper coil from the liquid N_2 Dewar. Power to the heaters for continuing the temperature rise of about 1°C/min. is handled by the program.

11. When any preset limit is reached, Max. Temperature, Max. Sample Length, or Max. Static Tension, the system will automatically halt.

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12. During the course of the experiment, each measurement will be displayed in terms of E, tan δ and Temperature for convenience of the operator.

13. After a run, data are handled by loading the Data Reduction Program into the calculator and allowing it to search the recorded tape for data.

CONCLUSIONS AND RECOMMENDATIONS

A working prototype system has been completed which achieved the following improvements:

Parameter	Manual Operation	Automatic Operation
Labor/hours/run	3 - 6	1/2 - 1
Resolution of tan δ measurement	~.003	.001
Tan δ range	.001 - 1.7	.001 - >3
Time for data reduction	3 - 5 hours	a few minutes (2 - 10)

An attempt was made to keep "hardware" at a minimum and take advantage of the flexibility of the calculator-computer which is part of the system. The program as presently written works quite well but will be improved and refined with experience.

Possible areas for future work and development might be:

a) Introduce servo-control (closed loop) on stress and/or strain amplitudes. This could extend the system to creep, stress relaxation and thermal expansion measurements.

b) Write programs for digital printer/plotter.

c) Develop hardware and write programs for automatic analog plotting of data.

COOLDOWN ROUTINE

This program is designed to maintain sufficient tension on the
sample to keep it straight as it is cooled. The tension control
senses buildup of force as the sample shrinks and the stepping motor
steps to relieve tension to keep within the limits. The program
constantly checks for excesses in any of the preset test limits
and for the preset minimum temperature. When the minimum or
starting temperature is reached, the display shows ...Minimum
Temperature... and the program halts. To start the test run,
strike CONTINUE.

COOLDOWN PROGRAM
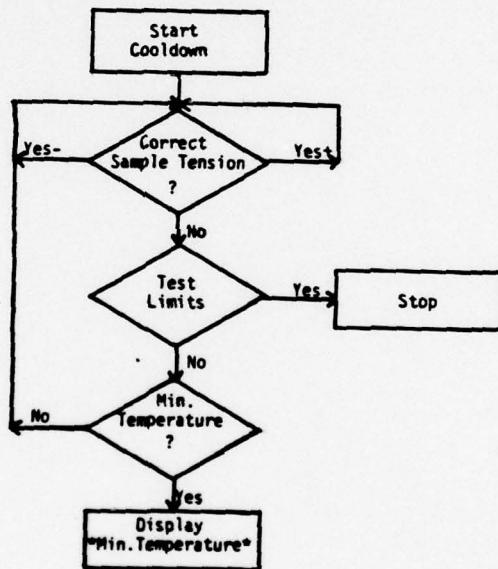
Figure 5

The main control program is
designed to measure and record
data as the temperature is in-
creased. It is a repetitive
process which starts with a
check on the size of the signal
from the load transducer for the
purpose of autoranging the lock-
in amplifier. After autoranging
the system reads the in-phase
and quadrature load signals and
stores them in the calculator
memory. The program then
branches into a subroutine to
measure static tension on the
sample and make any corrections,
+ or -, to keep it within proper
limits. It then proceeds to
measure and store the in-phase
and quadrature displacement
signals from the Rheovibron.
Next, the program makes a cor-
rection in sample length for
any + or - steps required to
correct the tension. That is
followed by another tension
measurement and correction, if
necessary. At this point the
temperature is measured and
stores. The program then makes
use of the scale and calibration
factors prestored in the memory
to calculate the modulus, E,
and tan δ. The values are
displayed with temperatures
so that the operator can follow
the course of the test. The
system then checks to see if
limits have been exceeded and,
if not, records the data on
tape. The process then repeats.

MAIN CONTROL PROGRAM
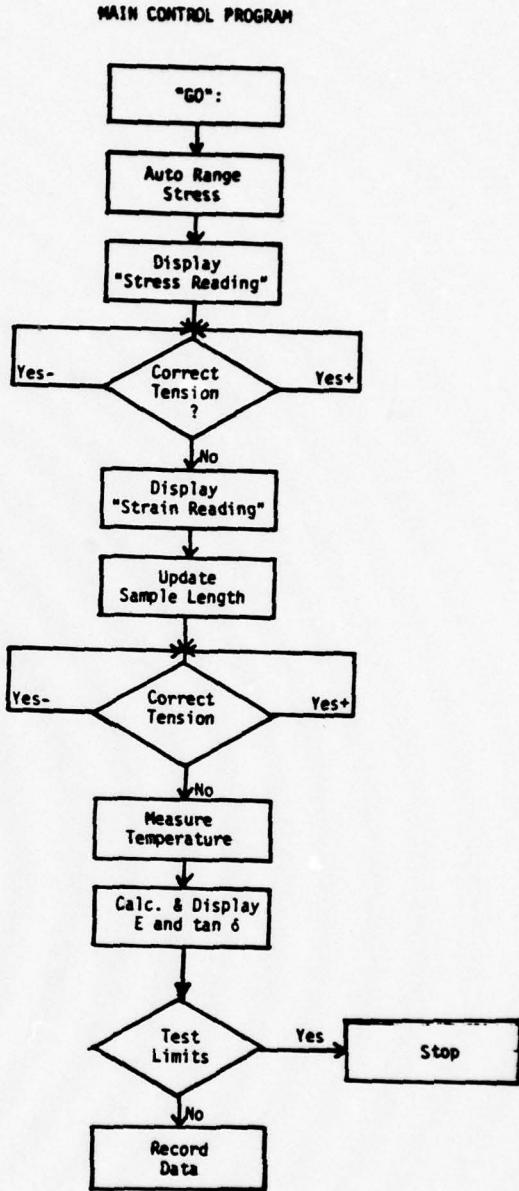
Figure 6

The data reduction program utilizes a standard curve fitting subroutine that smooths raw data by means of a 4th order poly-nominal regression. The first step in the program is to ask the operator to select a file number that specifies the header for the experimental data to be analyzed. The program will check to see if this file is a valid header file. This is accomplished by identifying the file and testing to see if the actual byte size is 160 -- the size of a header file. If not, a message is displayed and the program halts. If it is a valid header file, the processing continues.

The next step is the calculation of the sample volume from the input specifications. If the volume is zero, the program halts.

Next the program sets the initial temperature reference and the temperature limit which is the temperature reference plus ten degrees. This allows for smoothing over a ten degree range.

MAIN DATA REDUCTION PROGRAM

Figure 7A

12
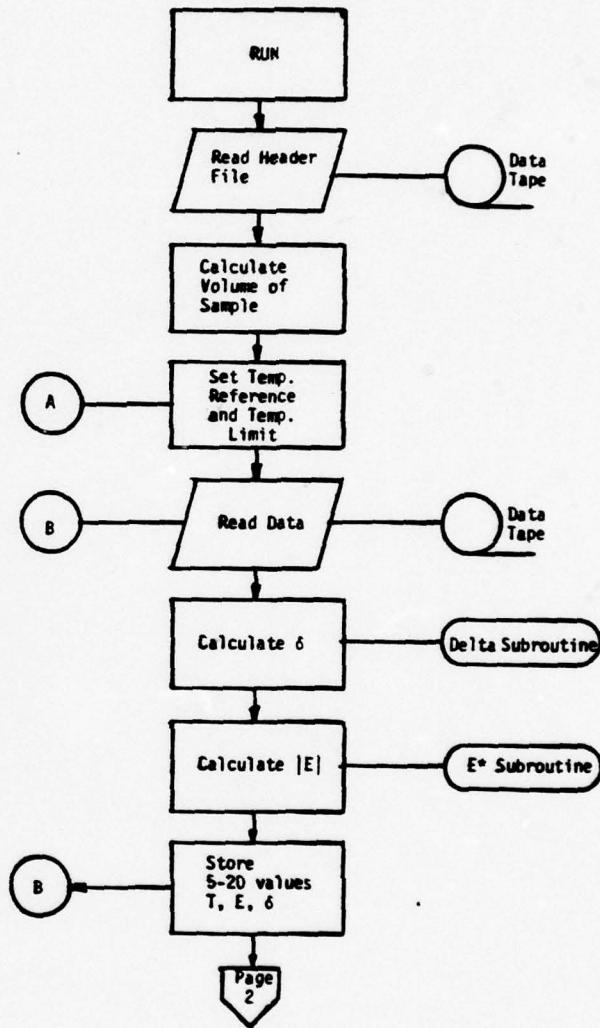
The program next reads the data sets, each consisting of a 7 element array containing the measured parameters needed to plot a tan delta curve. Young's Modulus and the angle, δ, are calculated from these data points and, along with the temperature, they are stored in arrays. When the temperature reading has exceeded the temperature limit for this curve the arrays are read into a subroutine "FIT2" which will calculate the compliance coefficient for the polynominal regression. These coefficients are then used in the equations to calculate "E" and tan delta. Before the next set of data prints are read from the tape the last five data sets in the arrays are stored into the first five locations of the next data sets. This will allow for a smooth progression. This process repeats itself for every ten degrees until the data runs out or until the maximum temperature has been reached.

Figure 7B

13
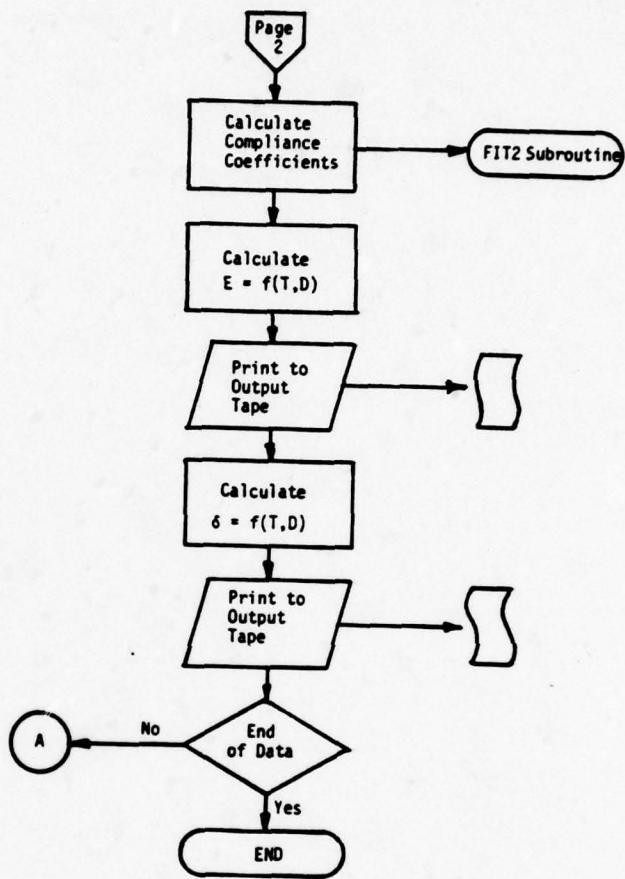
PARTS LISTS

DIAGRAMS

14
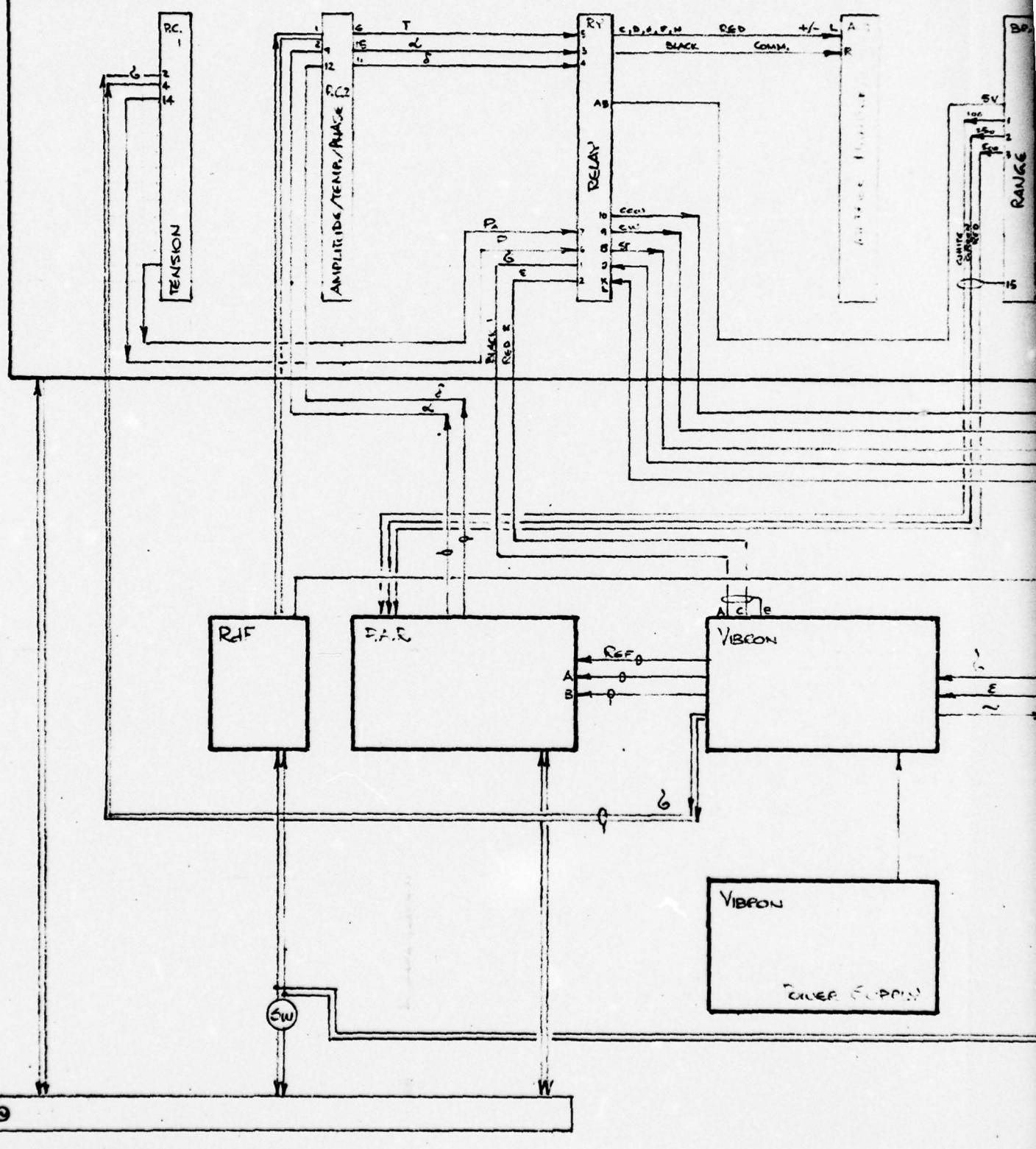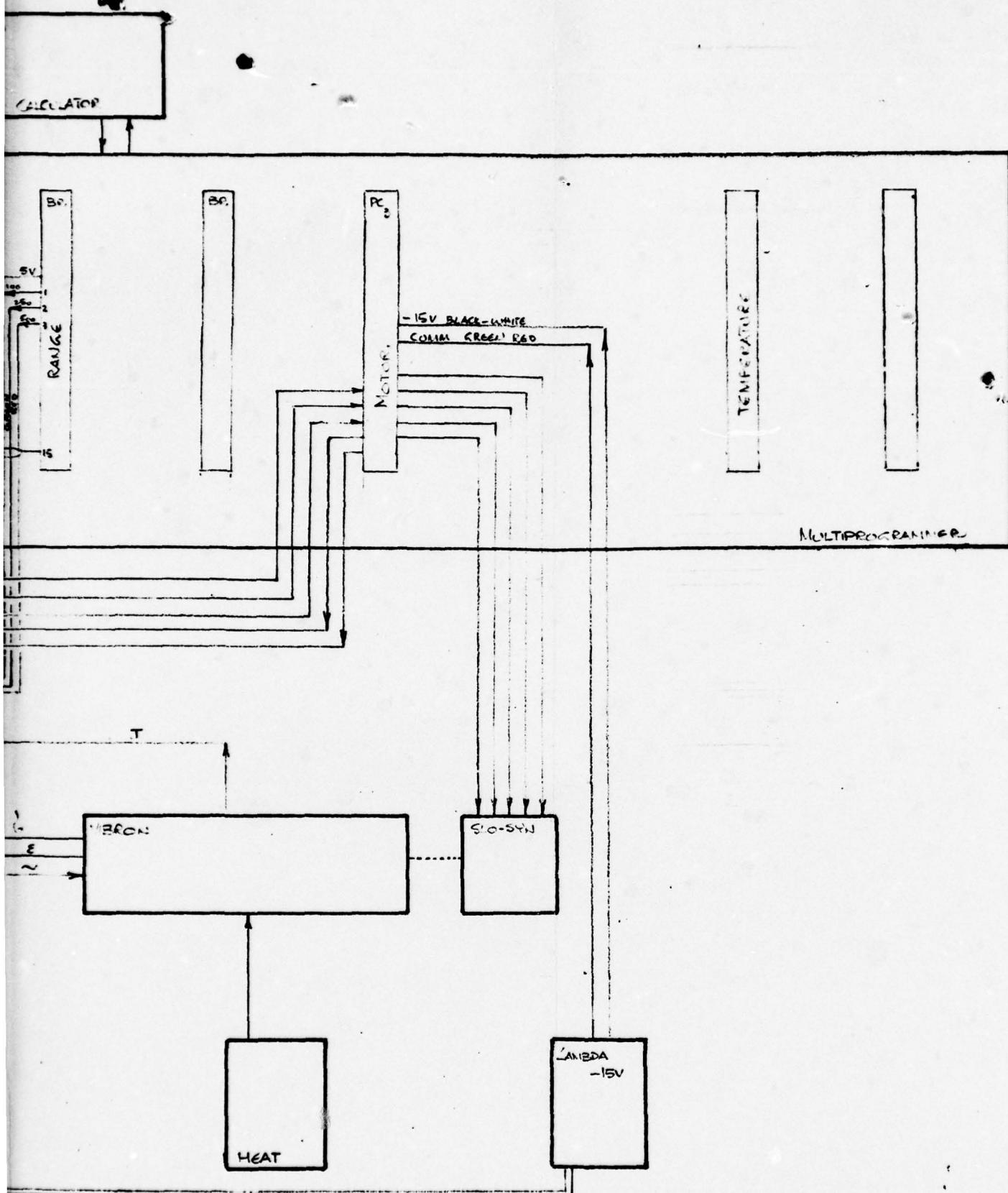
Overall Diagram of Interconnections

Ref.Desig.	Description	Q	Manufacturer
			Hewlett Packard:
CALC	Calculator	1	H.P.#9825A
ROM	9872 Plotter-General I/O Extended I/O	1	H.P.#98216A
MUL	Multiprogrammer	1	H.P.#6940B
	Voltage Regulator	1	H.P.#69351B
RELAY	Relay Output/Readback Card	1	H.P.#69433A
	Voltage Monitor Card	1	H.P.#69421A
PAR	Lock-In Amplifier	1	Princeton Applied Research #5204

CABINET

Ref.Desig.	Description	Q	Manufacturer
	Frame, Base & Casters	1	Equipto 40-3532-03
	Side Panel	2	Equipto 30-3538-11
	Top Panel	1	Equipto 20-2112-46
	Rear Panel, Louvered	1	Equipto 20-2812-14
	Equipment Shelves	2	Equipto 18-1925-01

Ref.Desig.	Description	Q	Manufacturer
	Stepping Motor	1	Superior Elec. SS50-1008
R1	Resistor, 5Ω, 25W	1	Superior Elec. BP262-G10
	Connector	1	Amphenol 3102A-14S-06P
	Mechanical Parts:		
	Housing		IDR
	Front Plate		IDR
	Shaft, Precision	1	PIC A3-30
	Bearing	2	PIC B10-9
	Collars	2	PIC C1-3
	Pulley	1	PIC FD6-64
	Pulley	1	PIC FD6-18
	Hub Clamp	4	PIC L4-6
	Drive Belt	1	PIC FA-95
	Motor Mount Plate	1	IDR
	Spacers - Housing	2	IDR
	Spacer - Motor Shaft	1	IDR
	Bellows Coupling	1	PIC T1-10
	Shaft, Precision	1	PIC A3-50
	1" Louvere	1	Midget Louvere Co.
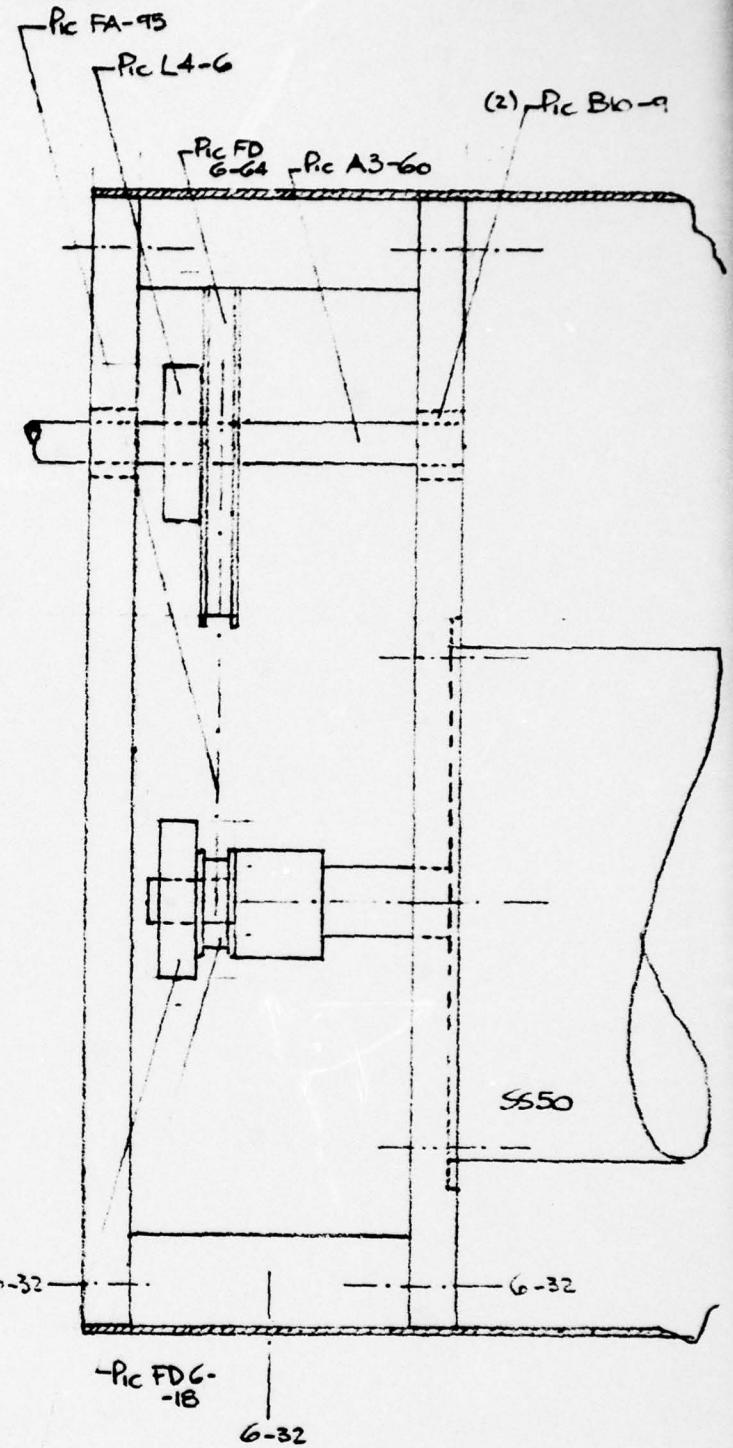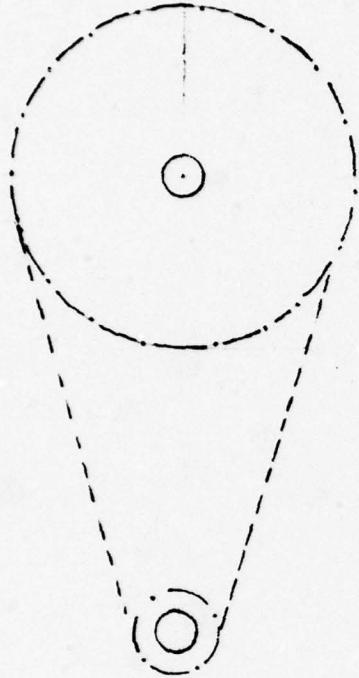
-Pic L4-6

Slo-Syn Drive Assembly

bR-Av₁-A118

DR-AV-A127
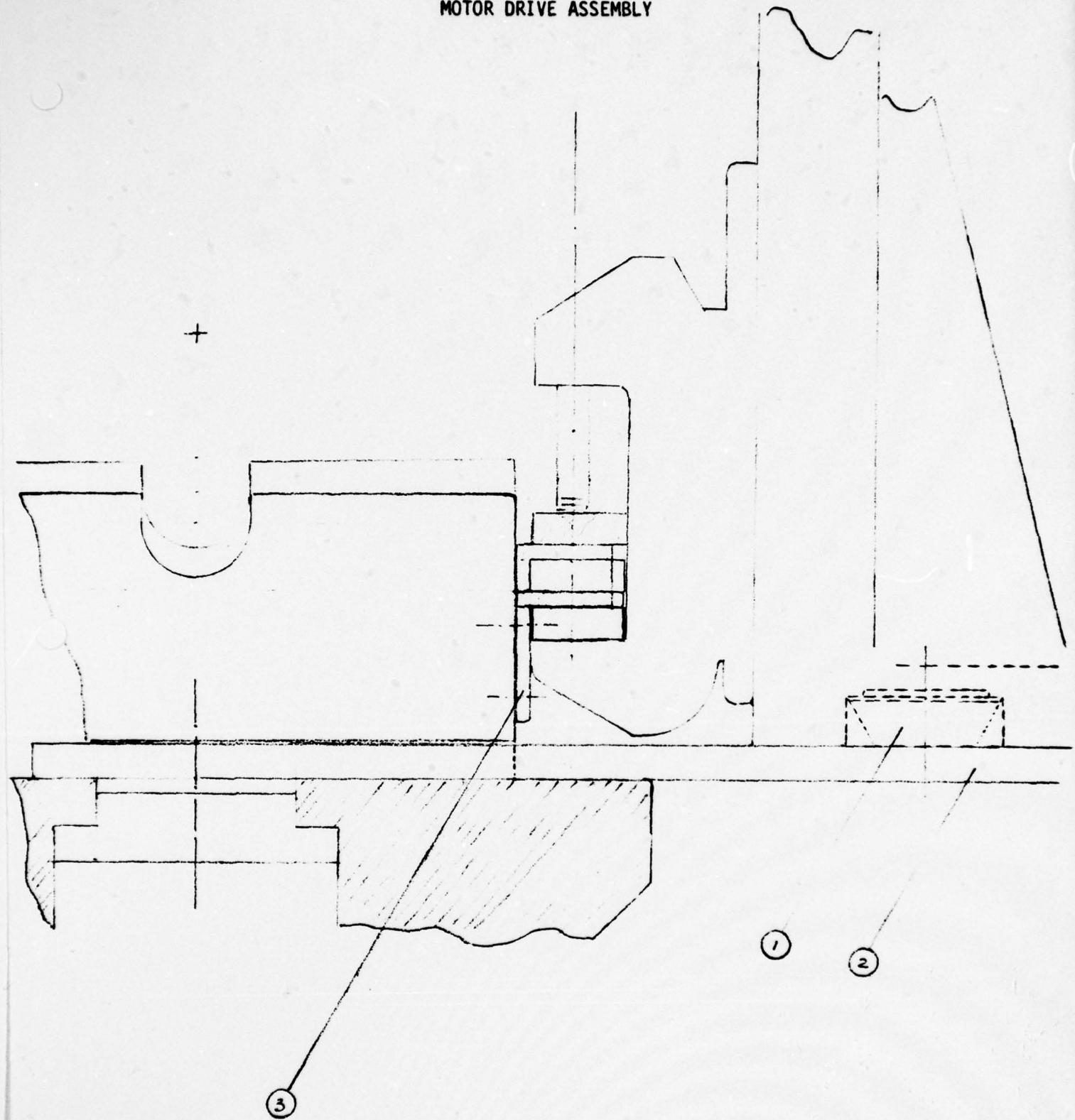
Motor
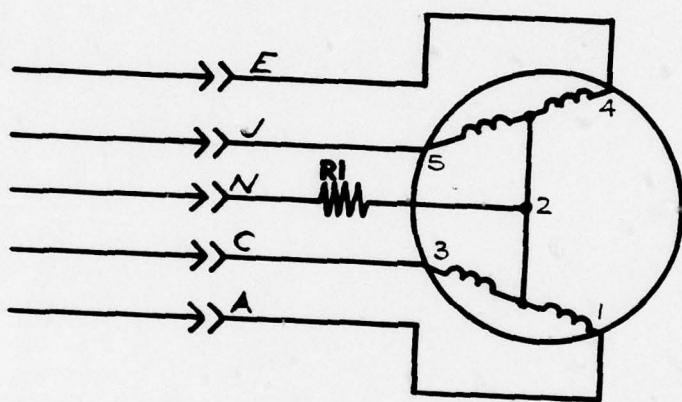
Ref.Desig.	Description	Q	Manufacturer
GPI/O	16-bit Duplex Interface	1	Hewlett Packard 98032A with opt. 040
RS232-C	Serial I/O Interface	1	Hewlett Packard 98036A
Dyn. Load	2 Conductor-Shielded	1	IDR #101
Dyn. Load- Displ.	3 Conductor-Shielded	1	IDR #102
Dyn. Dyn. Load, Load/ Disp., Ref.	BNC-BNC 6 ft.	3	IDR #103
I, Q	BNC-open 6 ft.	2	IDR #104
Range	5 Conductor Cable - Shielded	1	IDR #105
RTD	Sheathed Temp. Probe	1	RDF PN21701/110
Motor	6 Conductor Shielded	1	IDR #106
Power Strip	6 Outlet Wired Plug Mold	1	Equipto 03-0606-32
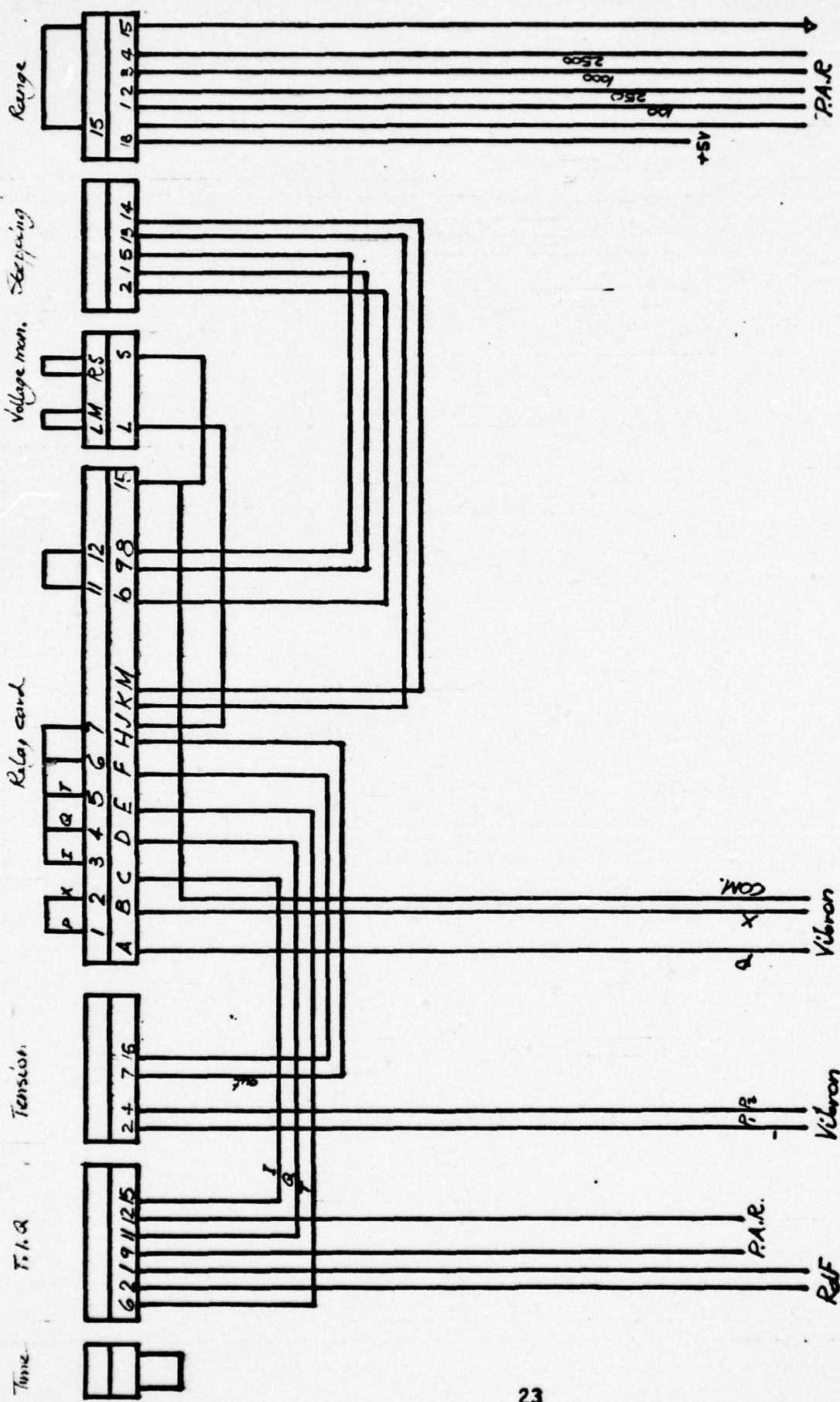
AMP.-PHASE-TEMP. Card

Ref.Desig.	Description	Q	Manufacturer
A1	Amplifier	1	Analog Devices 183J
A2, A3	Amplifier	2	Analog Devices 741H
R1	Trim Pot, 50KΩ	1	Allen Bradley RT5
R2	Resistor, Metal Film, 1.33KΩ	1	Corning C4
R3, R5	Resistor, Metal Film, 1KΩ	2	Corning C4
R4	Resistor, Metal Film, 200KΩ	1	Corning C4
R6	Resistor, Metal Film, 4.42KΩ	1	Corning C4
R7	Resistor, Metal Film	1	Analog Devices - Matched
R8, R10	Resistor, Metal Film, 2KΩ	2	Corning C4
R9, R11	Resistor, Metal Film, 1.2KΩ	2	Corning C4
R12, R14	Resistor, Metal Film, 10KΩ	2	Corning C4
R13, R15, R16, R17	Trim Pot, 10KΩ	4	Allen Bradley RT5
R18, R19	Resistor, Metal Film, 60KΩ	2	Corning C4
R20, R21	Resistor, Metal Film, 10KΩ	2	Corning C4
R22, R23	Resistor, Metal Film, 51KΩ	2	Corning C4
C1, C2	Capacitor, 5.0 MFD, 50VDC, ±5%	2	S&EI 22U
C3	Capacitor, 0.68 MFD, 50VDC, ±5%	1	S&EI 22U
	Mechanical Parts:		
	Connector 2x15	1	Hewlett Packard 251-15-30-261
	IC Socket 30 pin	2	Augat 8058-39G4
	Connector Jack	7	Cambion 450-3704
	P.C. Solder Terminal	50	Cambion 1461-2
	P.C. Board	1	IDR AV-100
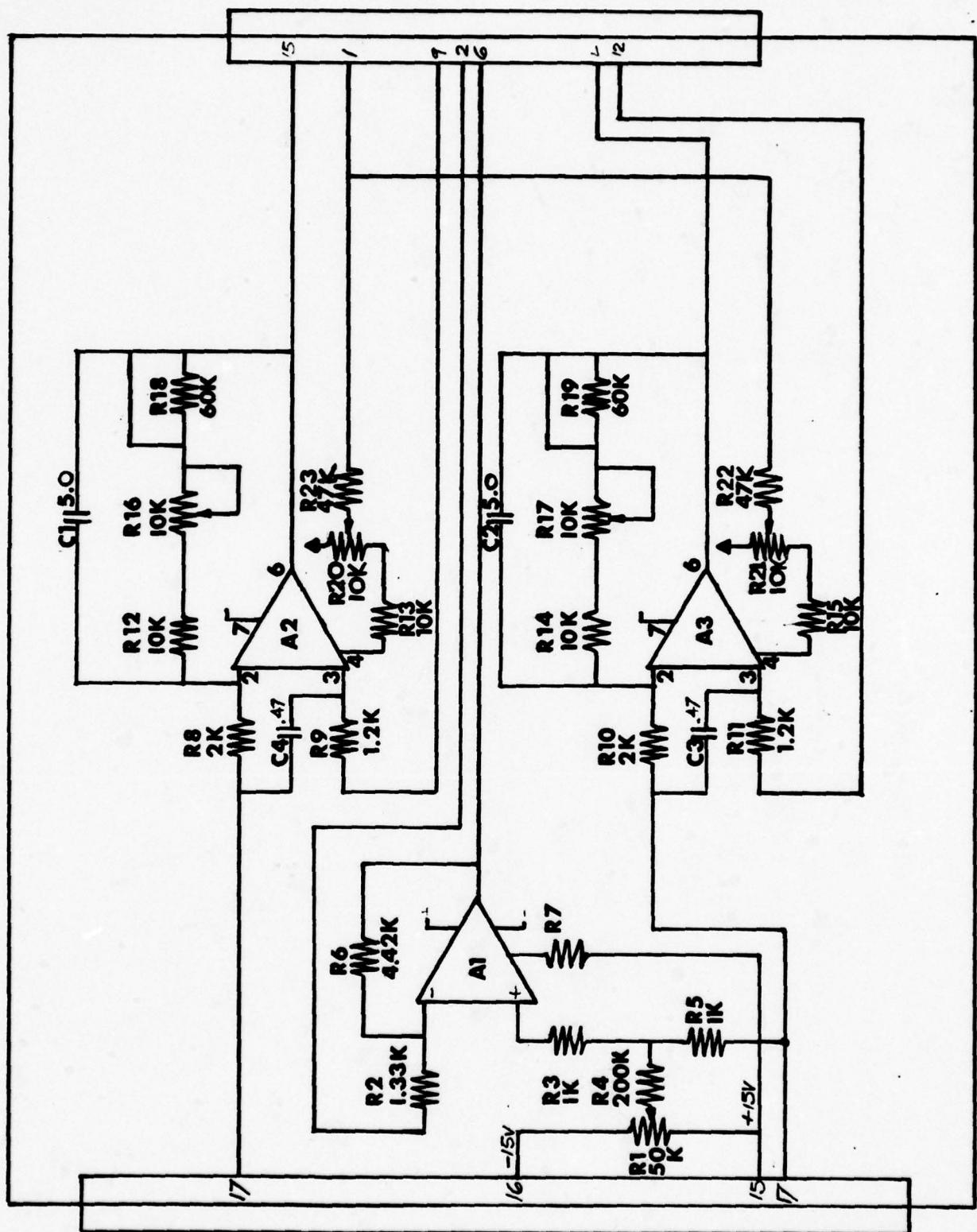
AMP.-PHASE-TEMP. Card
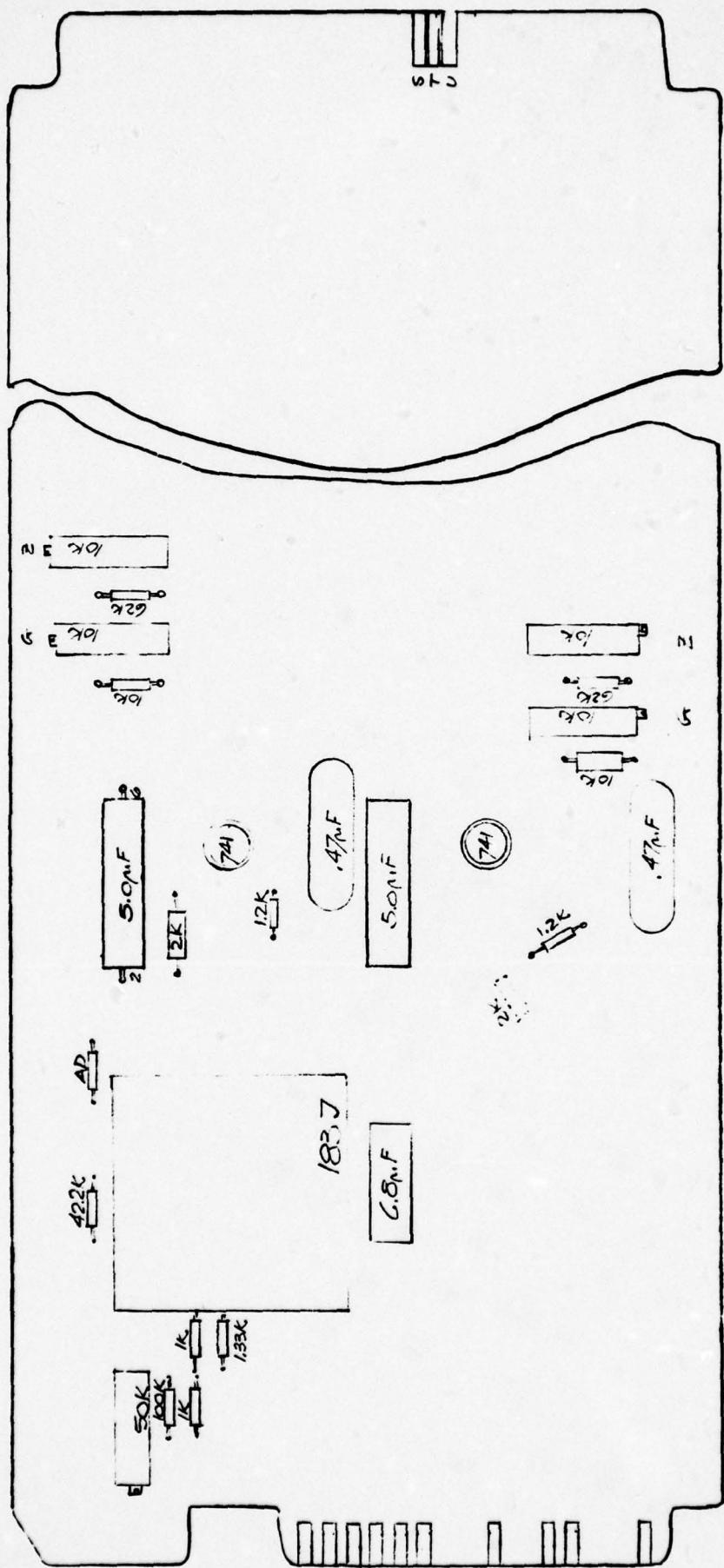
TENSION CARD

Ref.Desig.	Description	Q	Manufacturer
A1	Instrumentation Amplifier	1	Analog Devices 610L
R2	Trim Pot, 100KΩ	1	Allen Bradley RT5
R1	Resistor 402Ω 1%	1	Corning RN65D
	Mechanical Parts:		
	Connector 2x15 30 pin	1	Hewlett Packard 251-15-30-261
	P.C. Solder Terminals	13	Cambion 1461-2
	P.C. Board	1	IDR AV-101
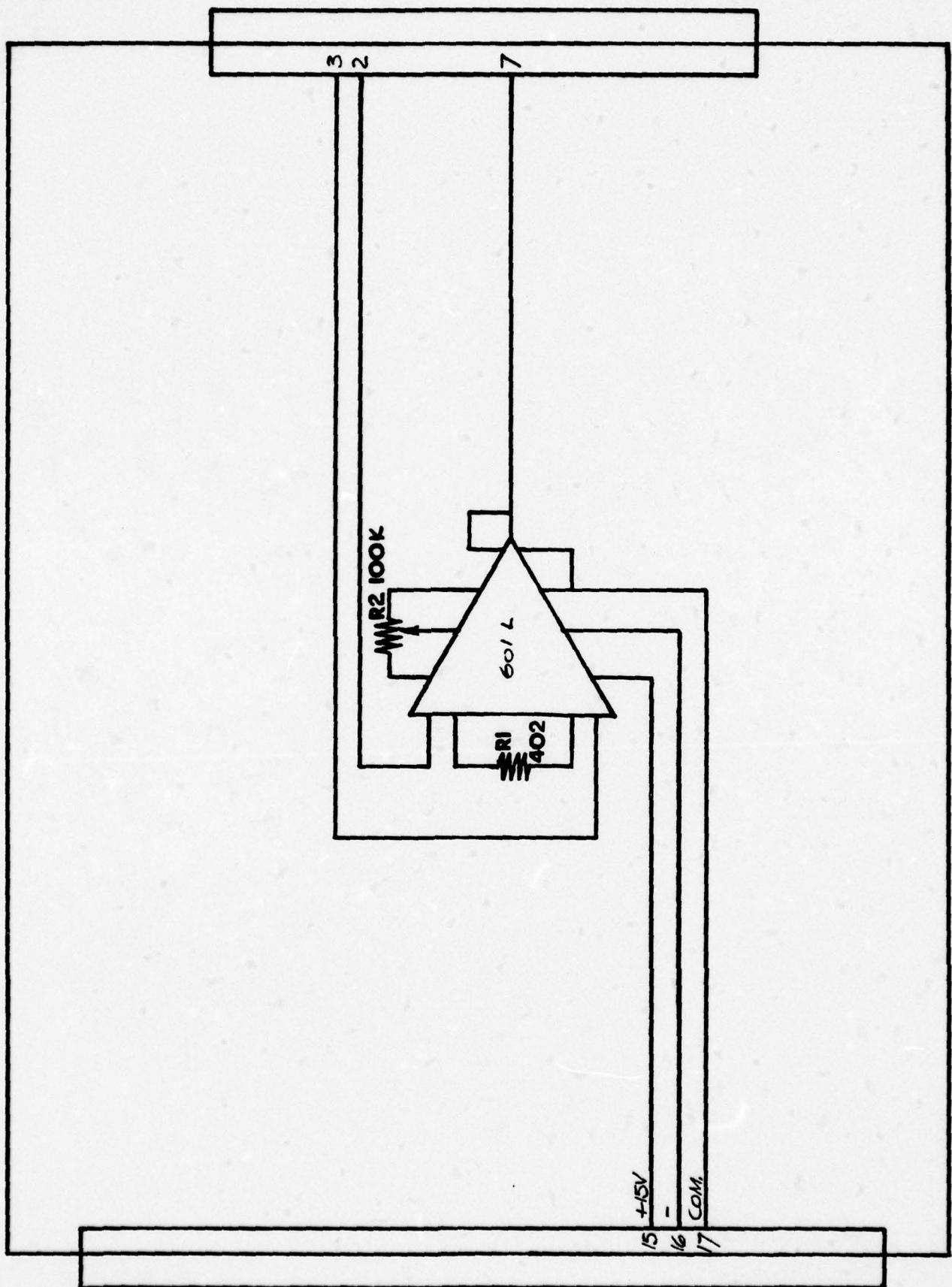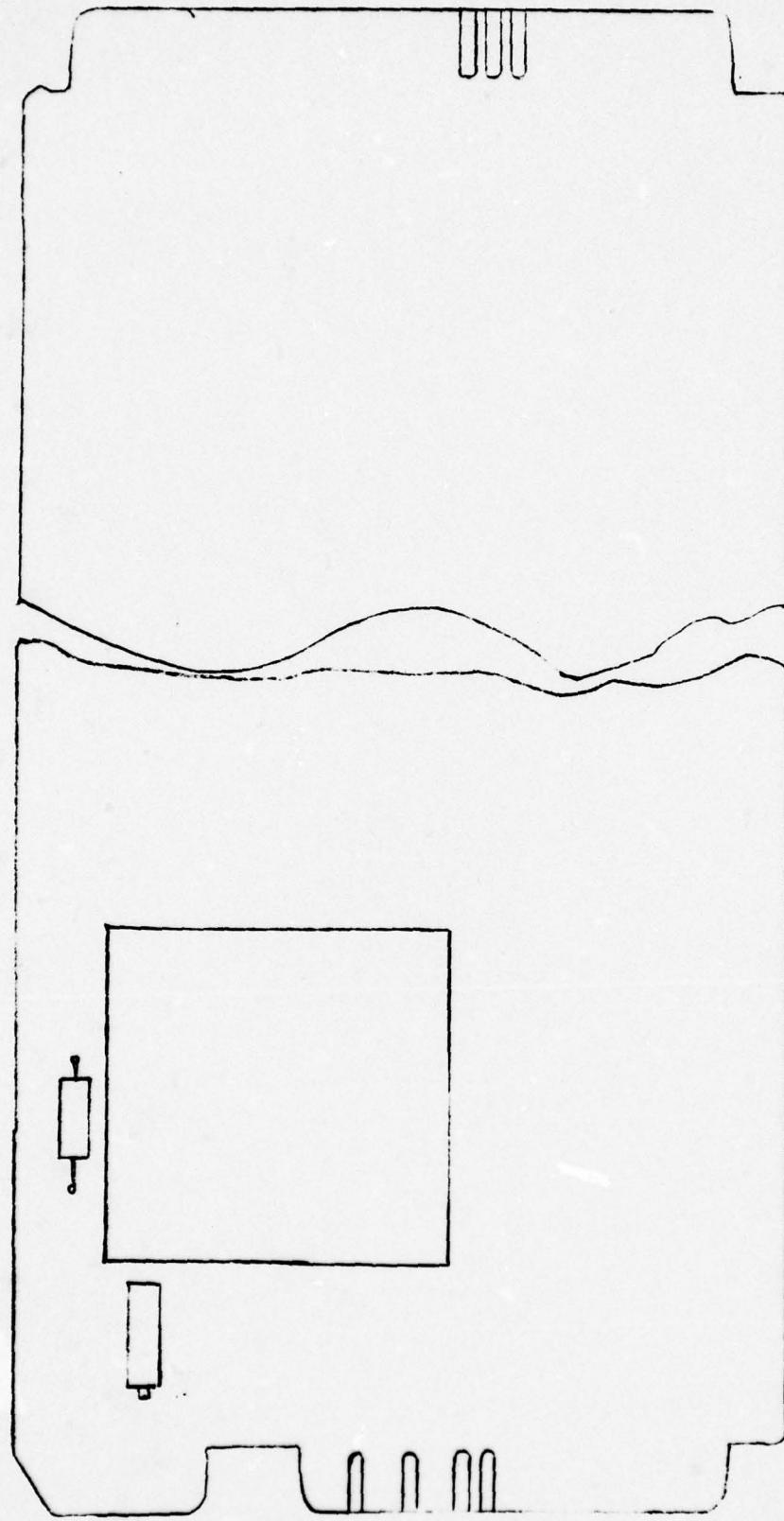
Ref.Desig.	Description	Q	Manufacturer
Motor	SLO-SYN Translator	1	Superior Elec. STM 1800
R1, R3	Resistor, Metal Film, 100KΩ	2	Corning C4
R2	Resistor, Metal Film, 47Ω	1	
R4	Resistor, Metal Film, 20Ω 10W	1	
C1	Capacitor 0.05 MFD±5% 50VDC	1	S&EI 22U
C2	Capacitor 0.1 MFD±5% 50VDC	1	S&EI 22U
	Mechanical Parts:		
	Connector 2x15 30 pin	1	Hewlett Packard 251-15-30-261
	Connector Jack	16	Cambion 450-3704
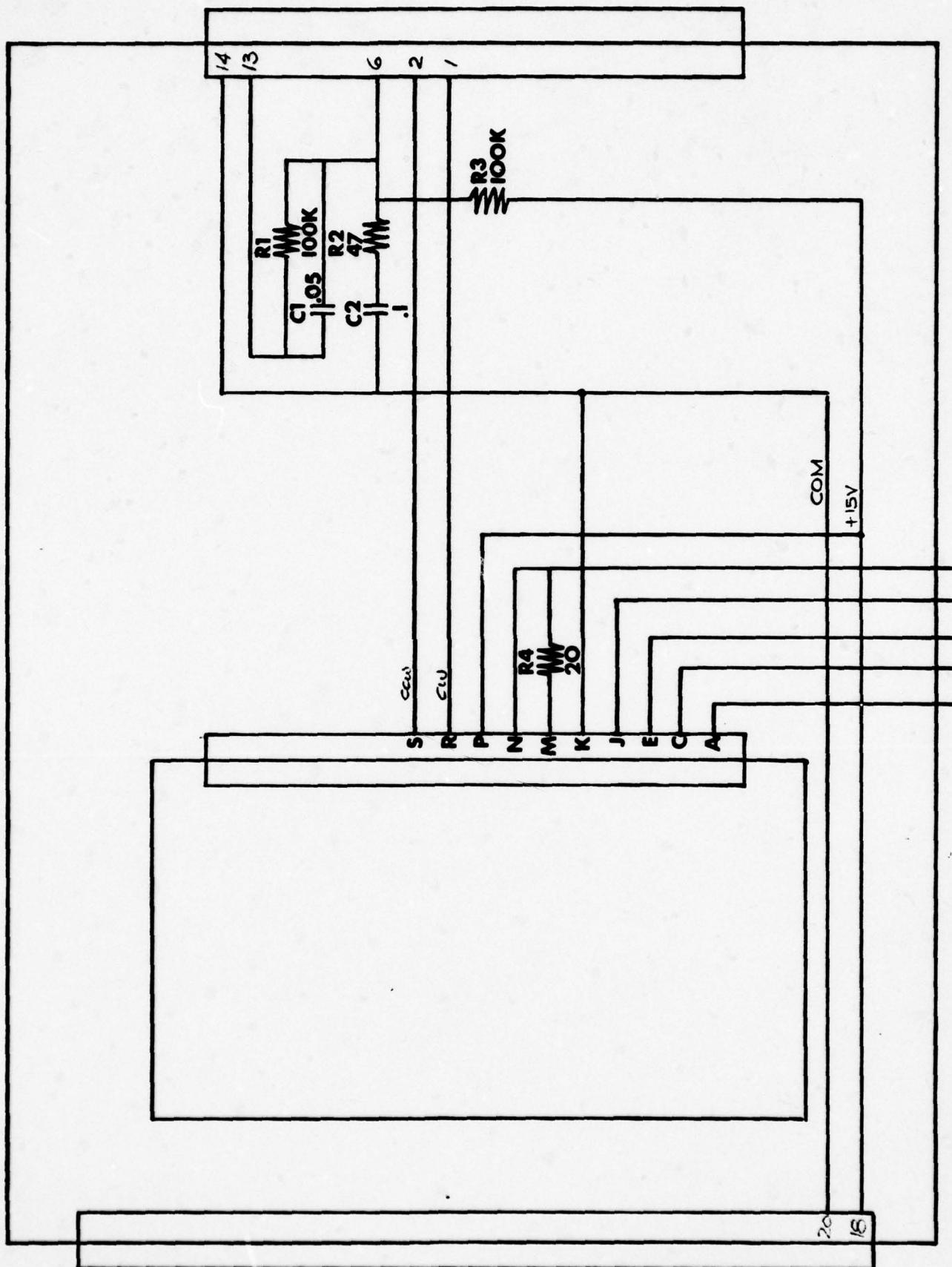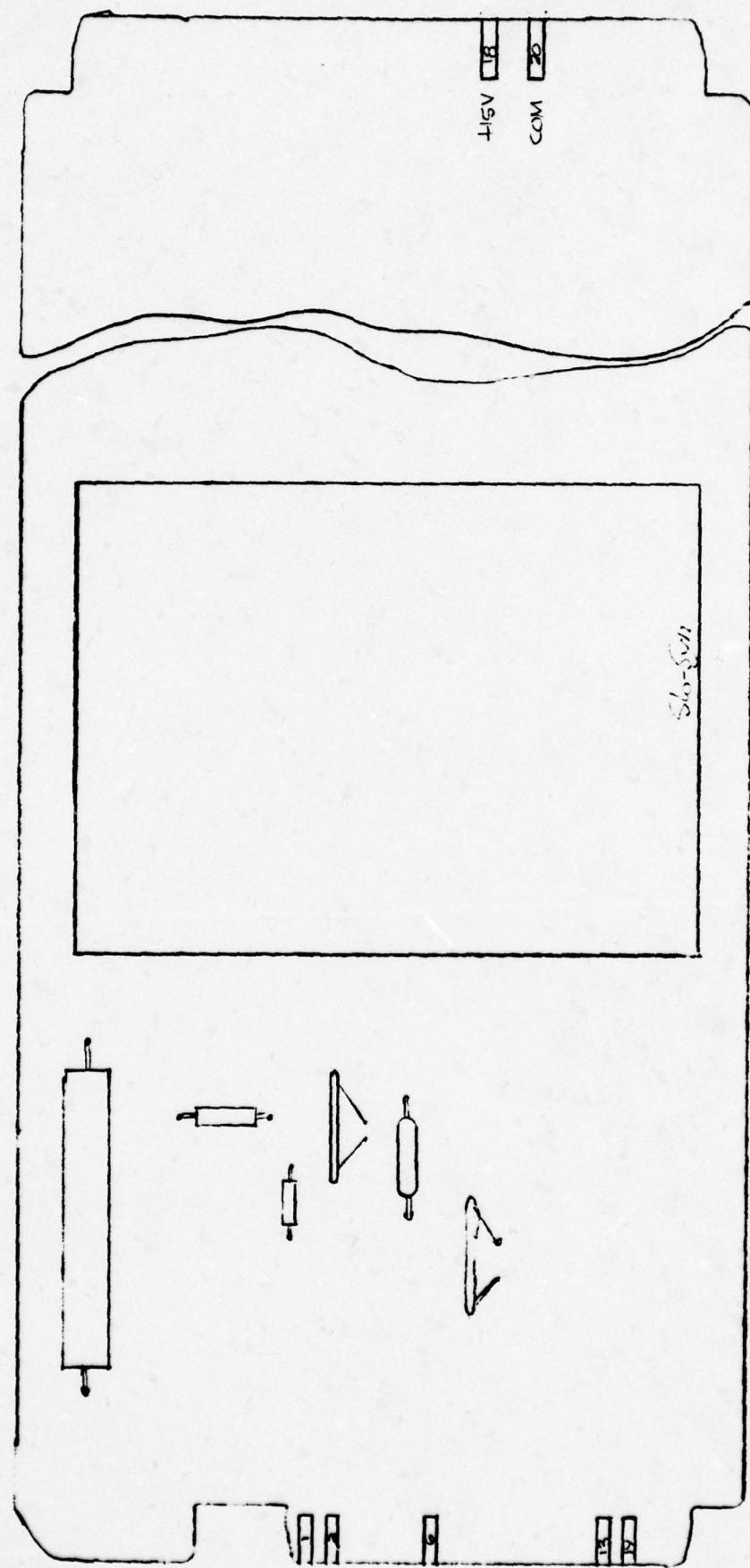
Ref.Desig.	Description	Q	Manufacturer
0. Bread-board	P.C. Board	1	Hewlett Packard 69380A
	In & P.A.R. Lock-In Amplifier		
K1,K2,K3,K4	Relay, SPST, 5VDC	4	Sigma 191TF1AZ-5S
R1,R2,R3,R4	Resistors, Metal Film, 10KΩ	4	Corning C5
Q1,Q2,Q3,Q4	Transistors	4	Motorola 2N4124
	Mechanical Parts:		
	P.S. Solder Terminals	25	Cambion 1461-2
	Epoxy-Glass Board	1	
	Mounting Bracket	1	
	Spacers	2	
	Screws	2	

33

34

Ref.Desig.	Description	Q	Manufacturer
	4.5" Long Vector Dip Plugboard	1	Vector Electronics 3662-5
K1, K2	Relay DPST	2	Magnecraft W171-DIP25
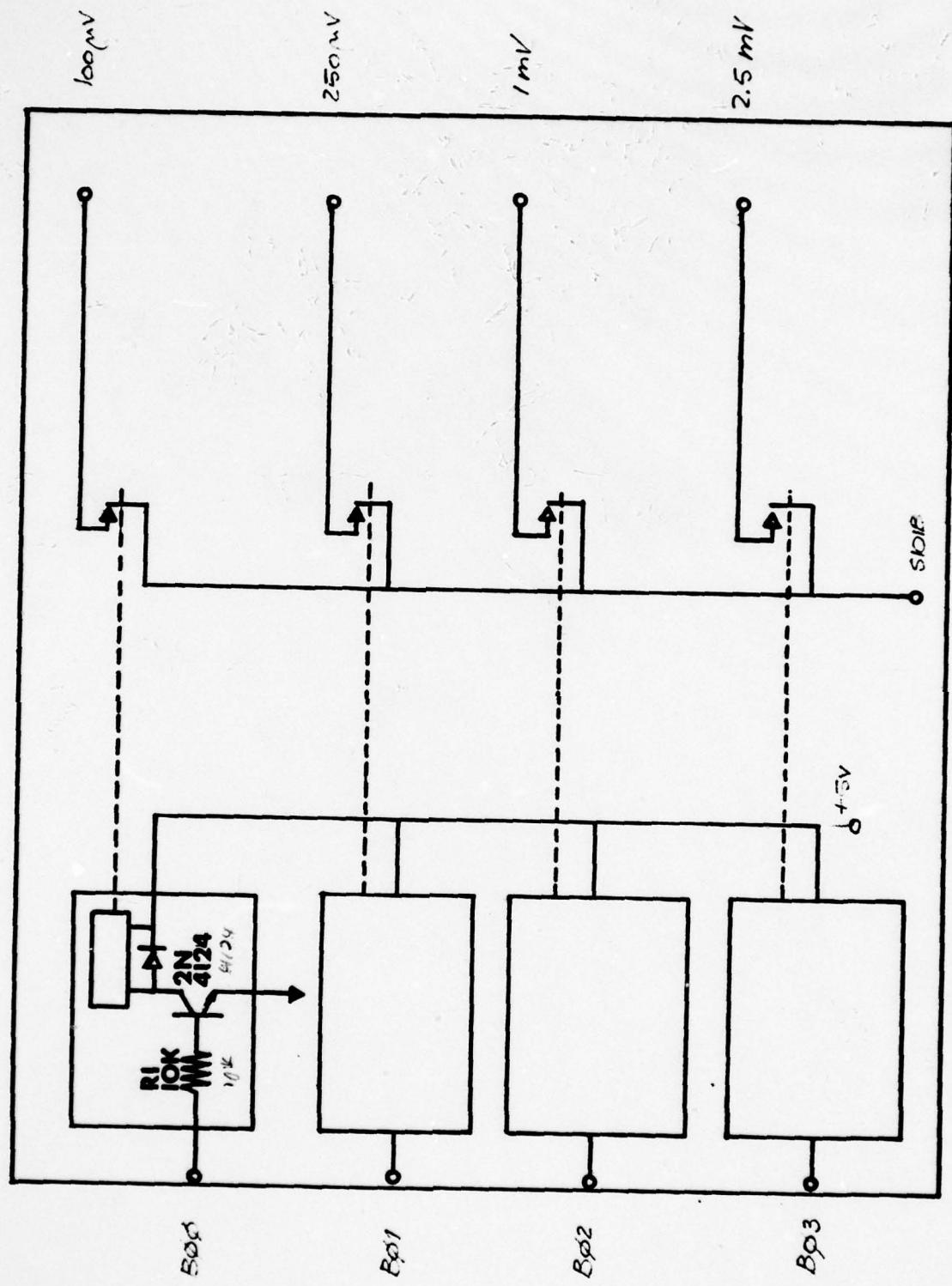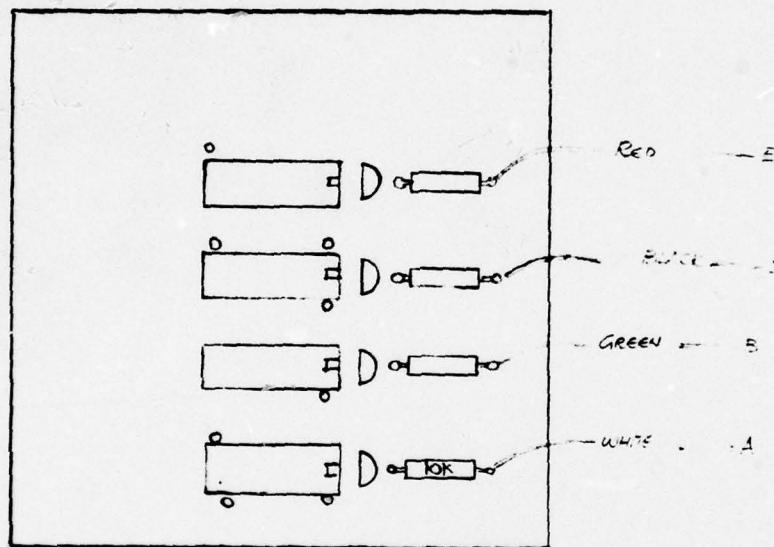
RELAY Board
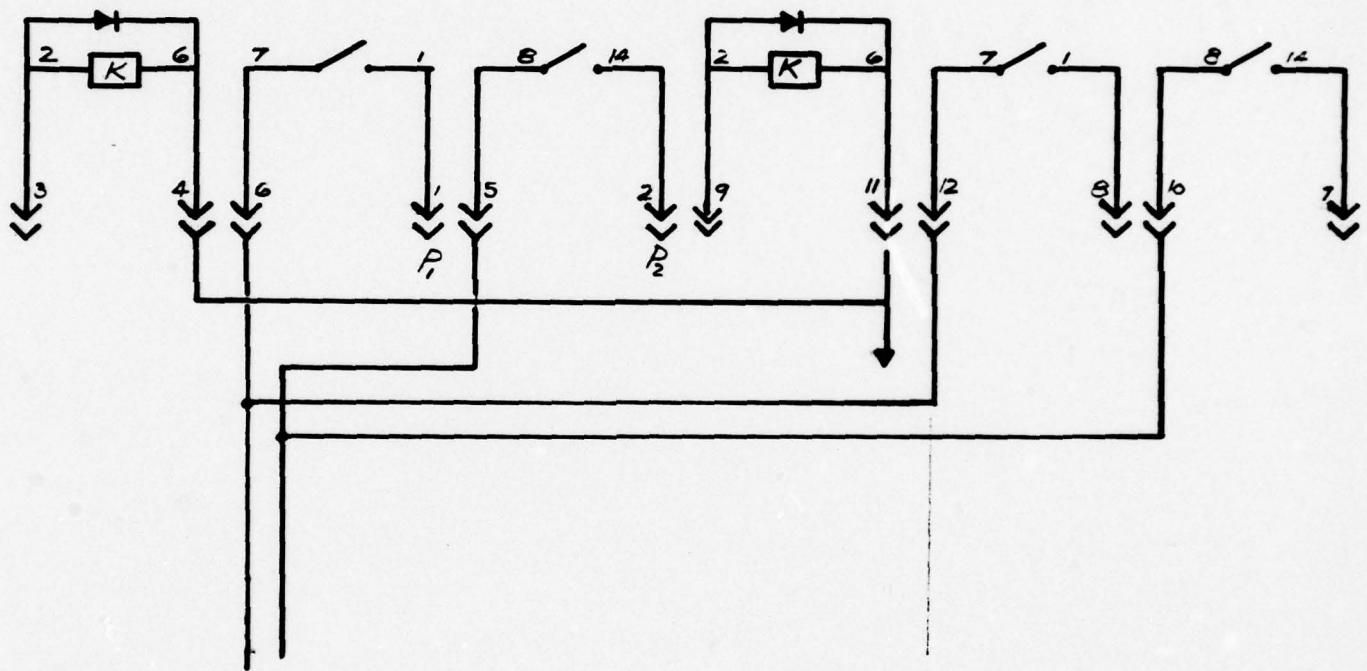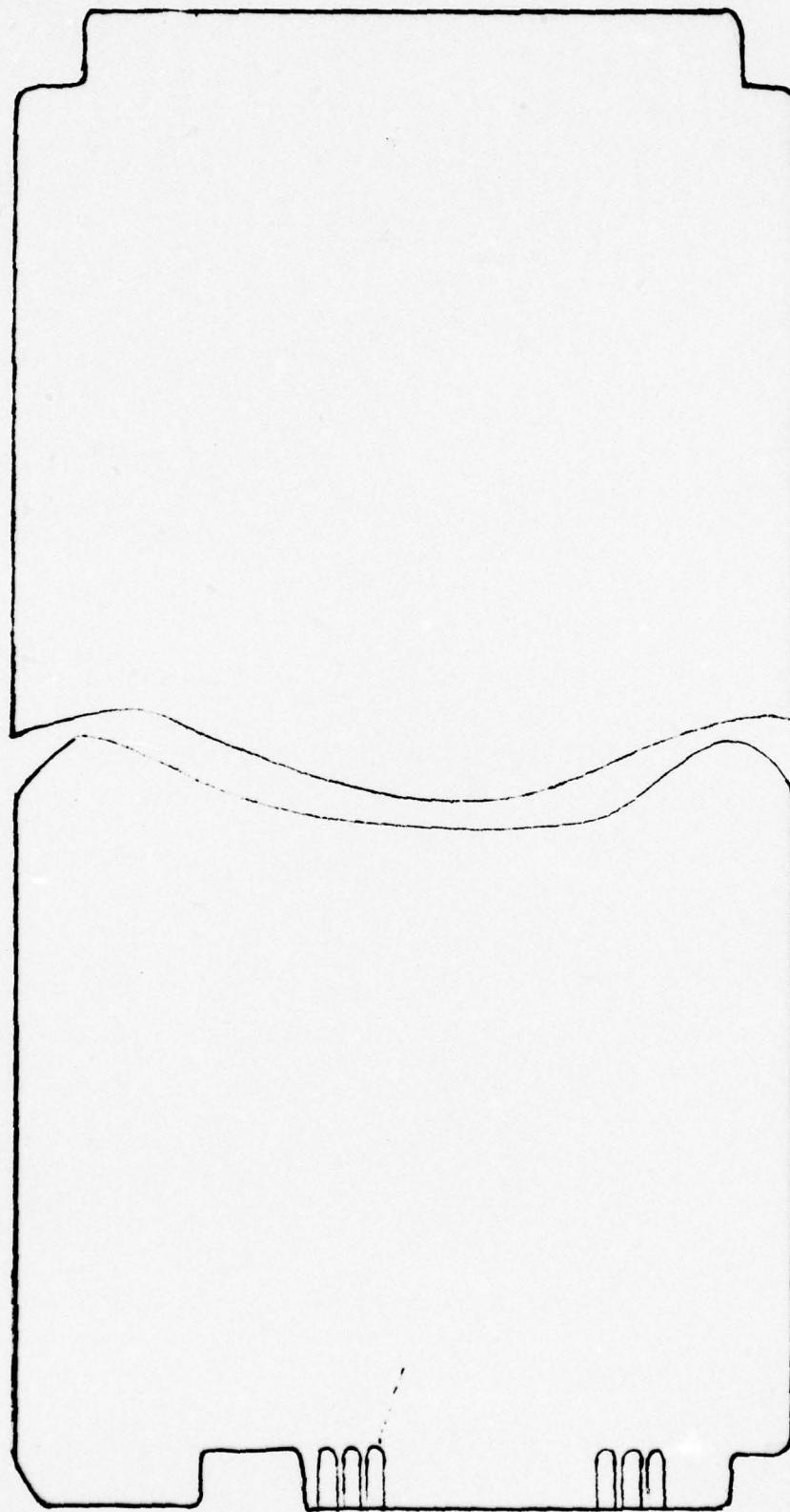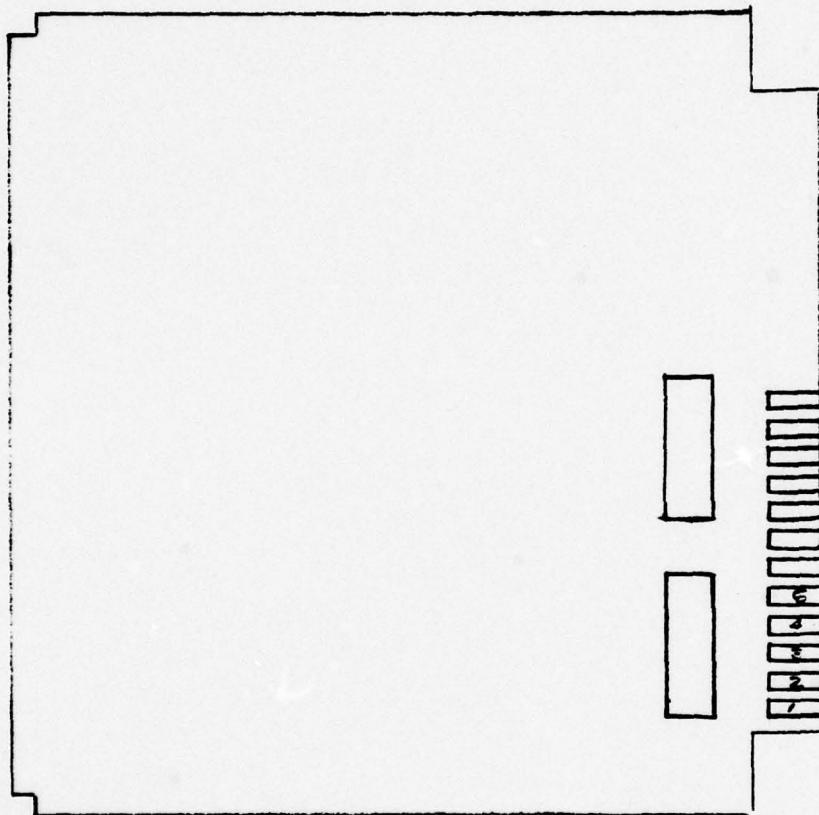
Ref.Desig.	Description	Q	Manufacturer
	Resistor, Metal Film, 100KΩ	1	Corning C4
	Resistor, Metal Film, 1.5KΩ	1	Corning C4
	Resistor, Metal Film, 10Ω	1	Corning C4
	Trim Pot 100K		Allen Bradley RT5
	Capacitor, 1MFD, 100V 5%	1	S&EI 22U
	Mechanical Parts:		
	Box 4" x 4" x 2"	1	Bud AU1083HG
	BNC Bulkhead Conn.	3	Amphenol 31-236
	Connector	1	Amphenol 3102A-10SL-03P
	Connector	1	Amphenol 3102A-12S-03P
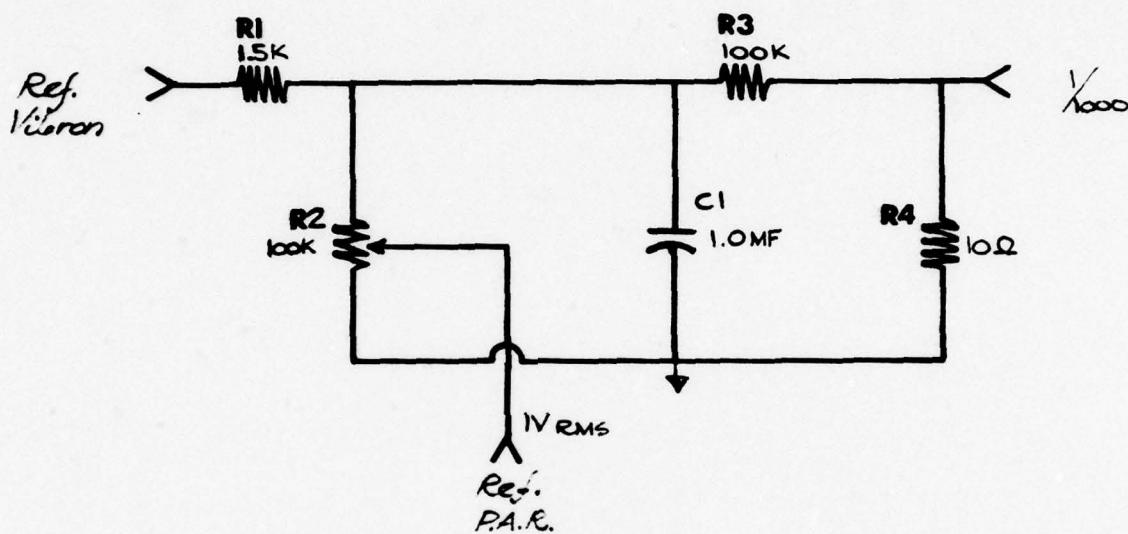
Ref.Desig.	Description	Q	Manufacturer
0. Bread- board	Breadboard P.C. Board	1	Hewlett Packard 69380A
K1, K2	Relay SPST	2	Magnecraft W107DIP5 or Sigma 191TE1AZ-5S
Q1, Q2	Transistors 2N3903	2	Motorola M2N3903
R1, R2	Resistor, Metal Film, 10KΩ	2	Corning C4

98380 A

2N 3703

SIGMA
191TE 1A2-5S

10K

R32

10K

RC1

10K

1 OA
2 OB (R02)
3 OC (RC1)

Signal driver
relays

44
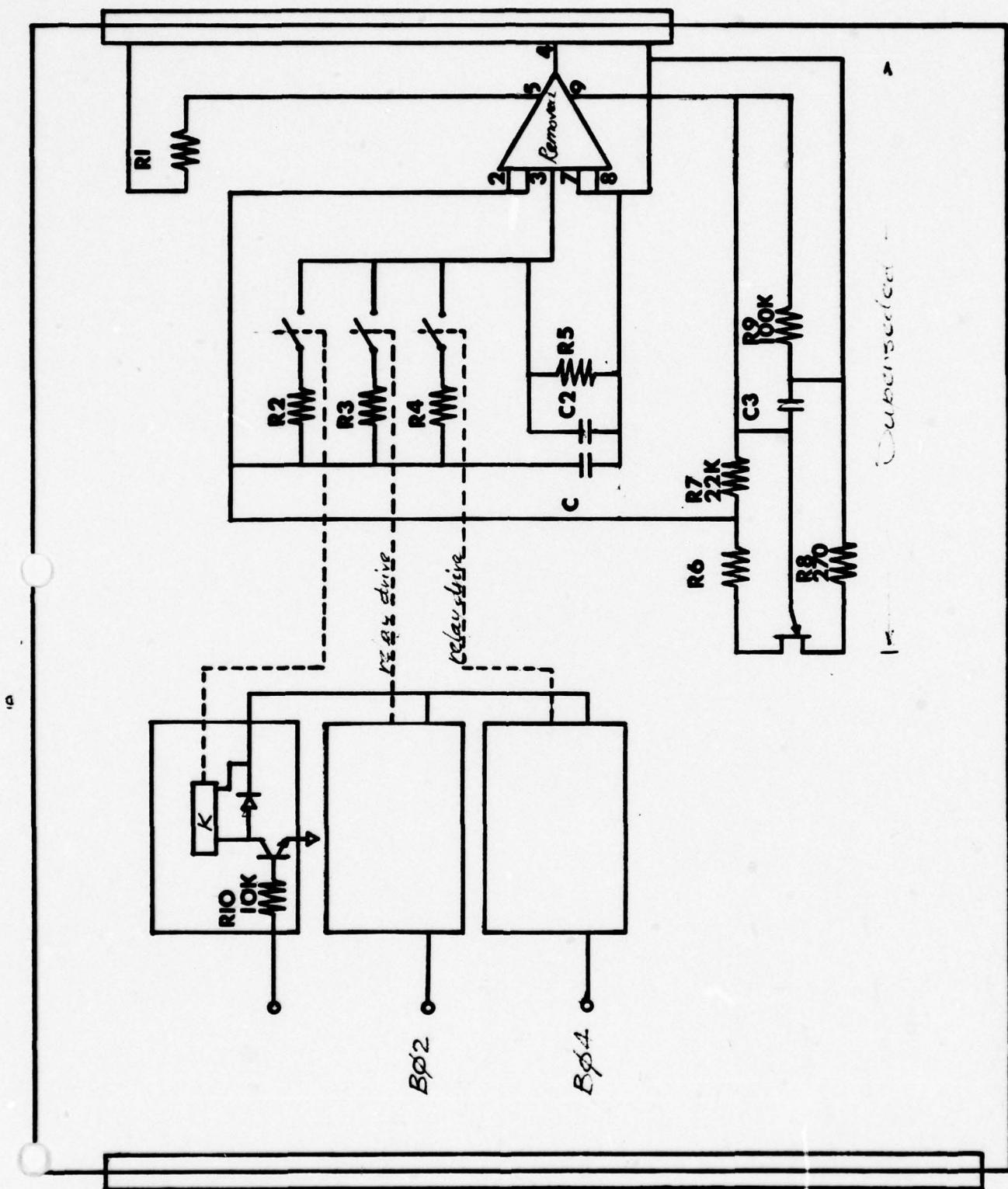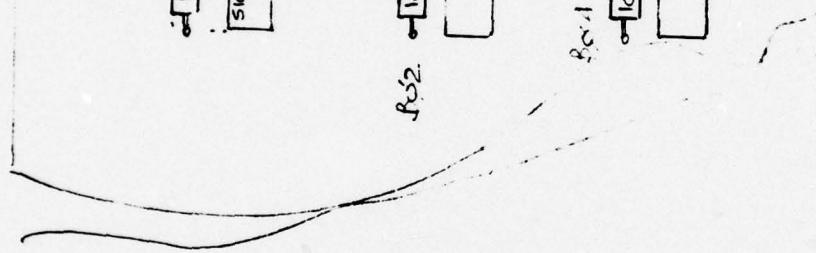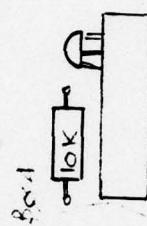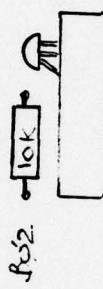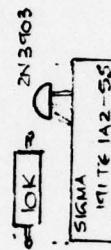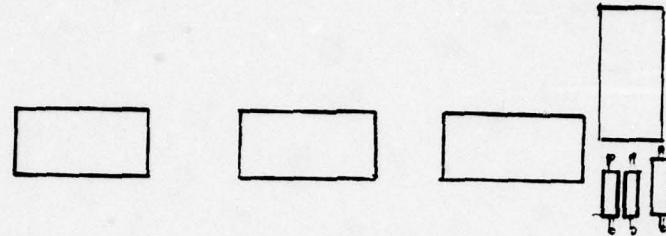
Temp.

POWER SUPPLY PANEL

Ref.Desig.	Description	Q	Manufacturer
	Power Supply	1	Lamda LOS-Y-15
K1, K2	Power Relay	2	Sigma 221A-1-5D
	Temperature Transmitter	1	RDF 2050-80RB
	<u>Mechanical Parts:</u>		
	Rear Panel	1	IDR #
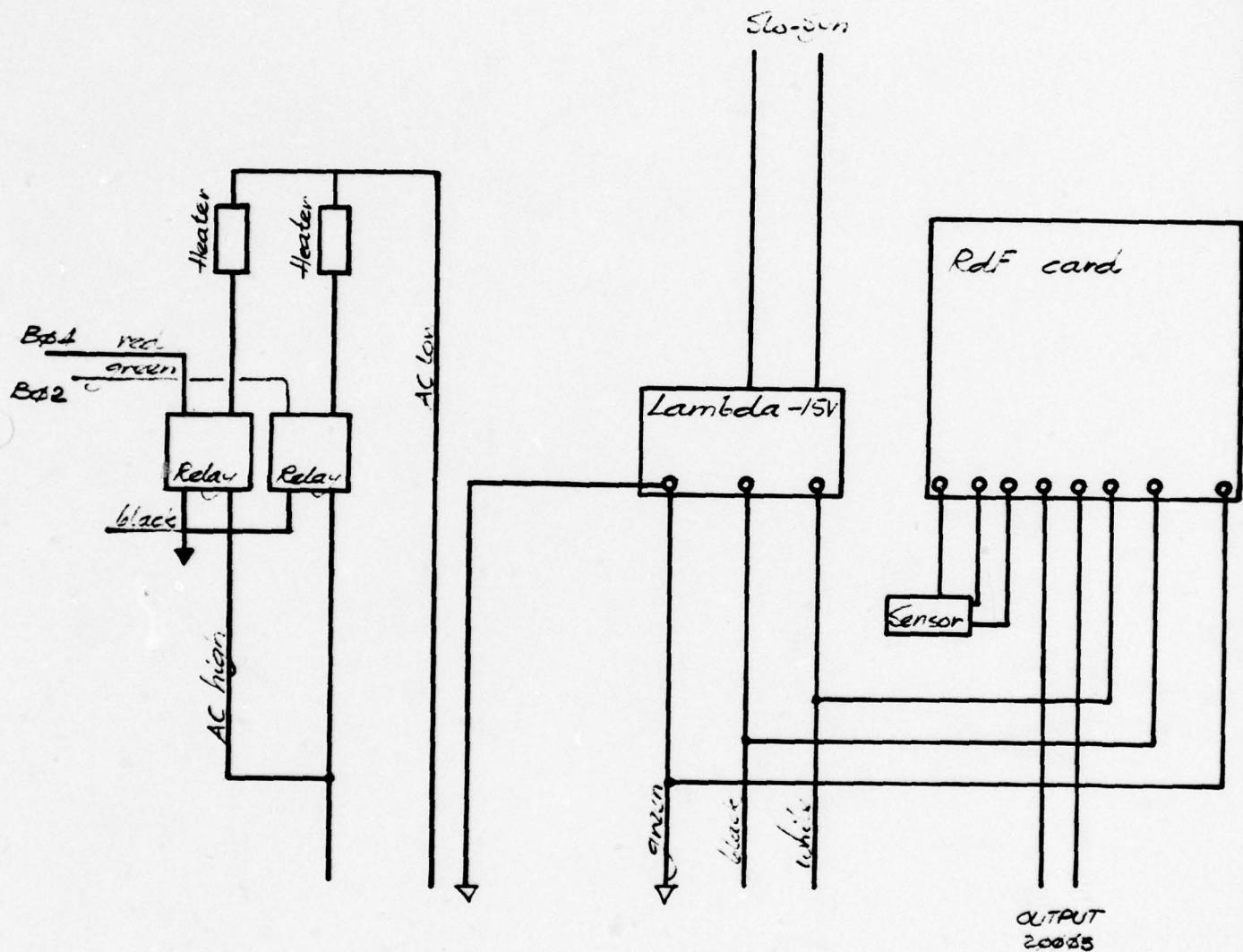
Ref.Desig.	Description	Q	Manufacturer
	Milling Attachment	1	Clausing M6-500
	Bottom Plate	1	IDR AV-A125
	Adapters	2	IDR AV-A126
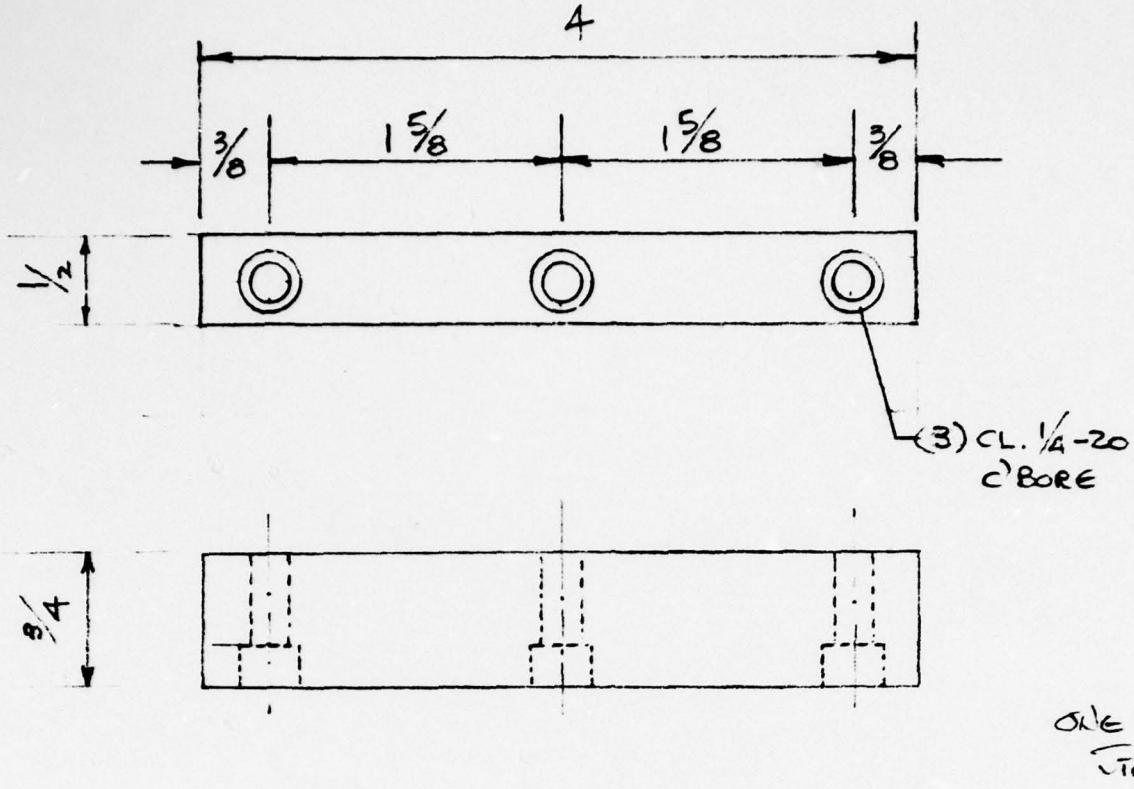
4

3/8   1⁵/₈   1⁵/₈   3/8

1/2

(3) CL. ¼-20
C'BORE

3/4

ONE REQUIRED
STEEL

Mounting for Temperature Chamber

60°

1.247ᴰ

.25ᴰ

ONE REQUIRED

48

IDR-IV-1126

DS-AV-A125

Mounting for Temperature Chamber

BASE

ONE REQUIRED

ALUMINUM
6061-T6
2024-T6

(3) 5/16 DIA

1/2   4½   4½   ½

1/4

1 3/8   5 3/4   1 3/8
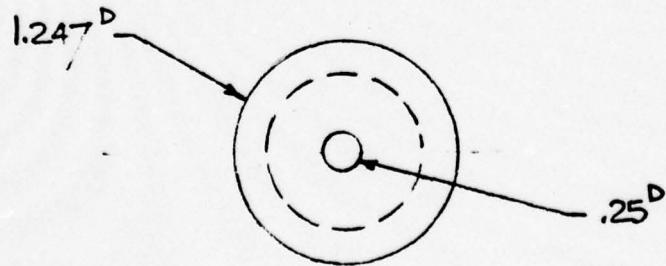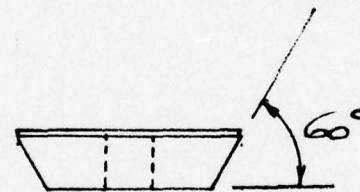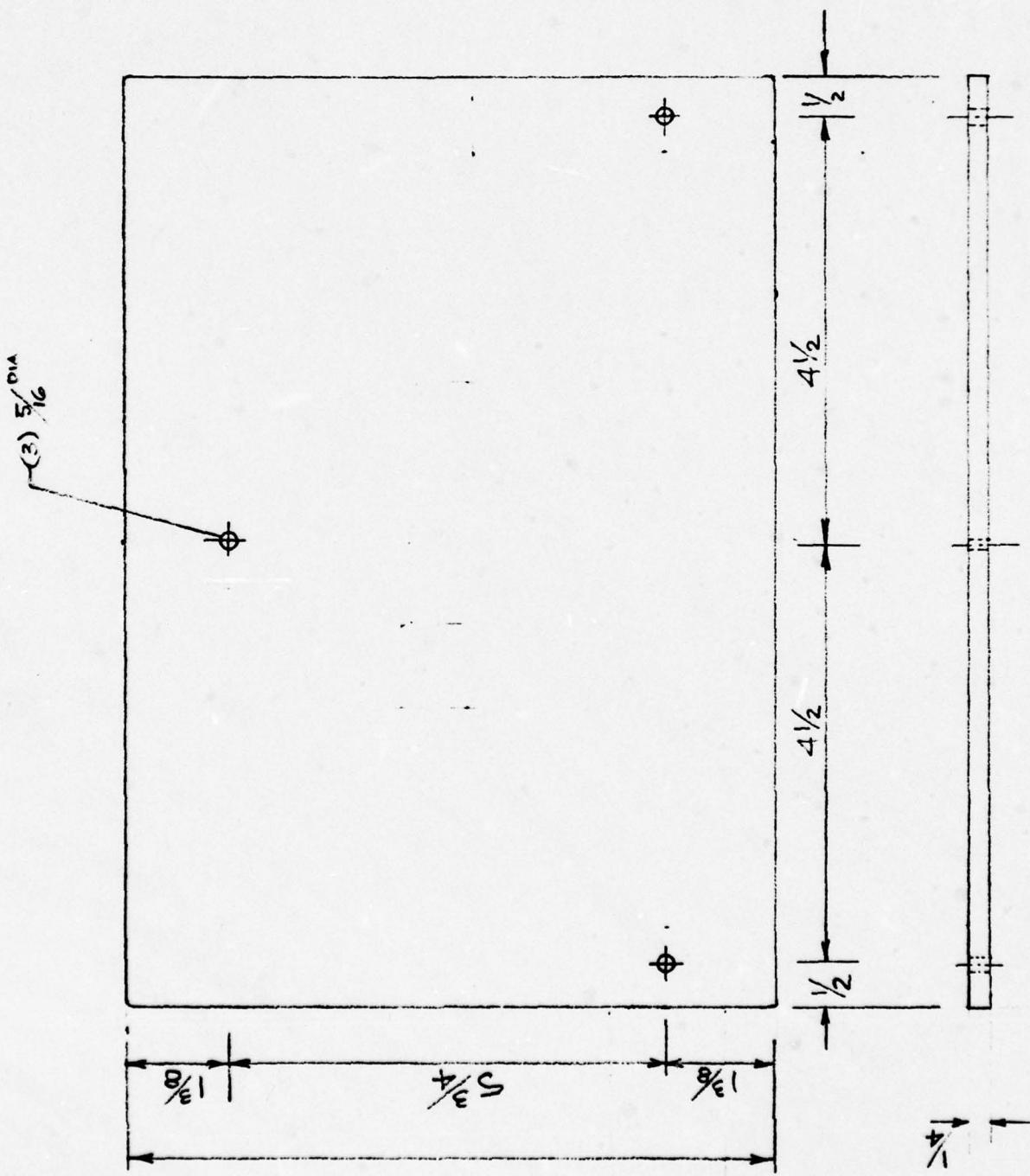
REFERENCES

1. Rheovibron Instruction Manual No. 62 (Nov. 1973)

2. Rheovibron Instruction Manual No. 68 (April 1973)

3. Rheovibron Maintenance Manual No. 68(70)-2 (June 1975)

4. Hewlett Packard 9825A Calculator Operating and Programming
   No. 09825-90000

5. Hewlett Packard 6940B Multiprogrammer Users Guide
   No. 59500-90005

6. Hewlett Packard Operating and Service Manual for Voltage
   Regulator Card, No. 69351-90002

7. Hewlett Packard Operating and Service Manual for Relay
   Output/Readback Card, No. 69433-90001

8. Hewlett Packard Operating and Service Manual for Voltage
   Monitor Card, No. 69421-90001

9. Hewlett Packard Operating and Service Manual for Breadboard
   Output Card, No. 69380-90001

10. Hewlett Packard Extended I/O Programming, No. 09825-90025

11. Hewlett Packard Installation and Service Manual for 16-Bit
    Interface, No. 98032-900006

12. Hewlett Packard Operating and Service Manual for Multiprogrammer,
    No. 06940-9000

13. Operating and Service Manual for Princeton Applied Research
    Lab. Lock-In Amplifier Model 5204, No. M5204; 8/77-GG

14. Hewlett Packard Installation and Service Manual for Serial
    I/O Interface, No. 98036-9-9000

PROCEDURES MANUAL

.

AUTOMATED RHEOVIBRON

CALIBRATION PROCEDURE

Using instructions contained in Rheovibron Manual #68, pages 14-18, balance, zero and calibrate both the strain and the stress gages.

Set up the instrument controls as follows:

> Bal-Calib Switch on Meas
> Main Switch on AF
> Tan δ Range Switch on 30
> AF Switch on 30
> DF Dial on 1000
> Amp. Adjust Dial fully counterclockwise
> Phase and Meas Switch on Meas
> Frequency Selector on any frequency above 1 Hz

Turn power on to the calculator and the multiprogrammer and load the program into the calculator memory. This is accomplished by inserting the mag tape cartridge, keying in fdfϕ, striking the EXECUTE key, and then keying in and executing the command to load fdfϕ. A pilot lamp next to the tape cartridge will light as the program is being loaded into the calculator. Run the "ZEROES:" program by keying into calculator the command RUN 210, striking the EXECUTE key. When the calculator requests a Z, key in number 6, Continue. A number will appear on the display which will represent in-phase component of the output of the strain gage (X_I). This should be zero (±0.005 volts). If it is not, refer to the instructions for removing offsets from the system.

When the in-phase channel is satisfactorily zeroed, repeat the program by keying RUN 210 into the calculator and striking EXECUTE. When a Z is requested, key in 12, Continue. You will now read the quadrature component of the strain (X_Q) and it, too, should be zero (±0.005 volts).

With no sample in the grips, gradually turn the Amp. Adjust knob clockwise until the tan δ meter registers exactly full scale. Again run the "ZEROES:" program by keying Run 210, EXECUTE. Key in a 6 for Z.

The number which appears on the display represents the magnitude of the in-phase strain component, X_I. Record it. Next rerun the program by keying RUN 210, EXECUTE, and providing a 12 for the Z. The number which now appears on the display represents the quadrature component of the strain transducer, X_Q. From these two values, X_I and X_Q, we can calculate the magnitude of the output of the strain transducer under these conditions:

i
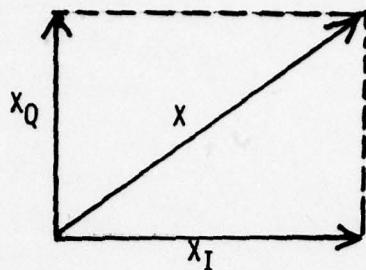
$$|X| = \sqrt{X_I^2 + X_Q^2}, \text{ volts}$$

Figure 8

The actual dynamic displacement amplitude measured by the transducer is exactly 5.0×10^{-3} cm. The calculation is explained on page 6 of the Rheovibron Manual #68. $|X| \div 5.0 \times 10^{-3}$ = scale factor for strain transducer, V./cm. This is the strain transducer calibration which is stored in the program.

Use the same procedure for the stress transducer except that a sample must be placed in the grips and sufficient tension applied to eliminate buckling of the sample when dynamic displacement is applied. The sample material is not important. The instrument controls are set the same as the previous measurement except that the main switch is now on DF instead of AF.

Gradually turn the Amp. Adjust control clockwise until the tan δ meter reads exactly full scale. The in-phase component can now be read by keying in and executing RUN which will initiate the "ZEROES:" program. Provide a 5 for the Z which will display the in-phase component of load, P_I. The quadrature component is determined by again running the program and providing an 11 for Z. The calculation is the same:
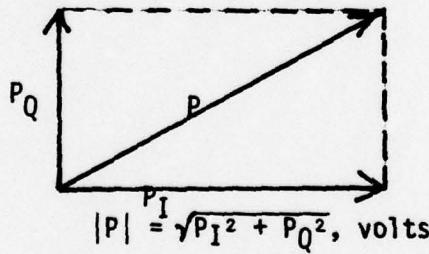
$$|P| = \sqrt{P_I^2 + P_Q^2}, \text{ volts}$$

Figure 9

The load signal is now exactly 10^4 dynes and the determination is again explained in Rheovibron Manual #68, page 6.

$$\frac{|P|}{10^4} = \text{scale factor for the force transducer, V./dyne.}$$

This is the stress transducer calibration factor which is stored in the program.

ii

PROCEDURE FOR CHECKING AND ADJUSTING SYSTEM ZEROES

1. Turn on the system.

2. Turn Amplitude Adjust fully counterclockwise.

3. Load the control program into the calculator.

4. Run the "Zeroes:" subroutine, RUN, EXECUTE.

5. When the "Z" is requested by the subroutine, enter 5 and strike "continue." This command will cause the system to switch on the stress signal and read the in-phase component P_I. The signal should be 0.000±0.005 volts. Record the value.

6. Strike RESET, CONTINUE, which will cause the system to request another Z. Type in 6 and strike CONTINUE. The output of the in-phase strain component, X_I, will be displayed. It, too, should be 0.000±0.005 volts. Record the value.

7. Repeat the same procedure using

"Z"	channel displayed
11	quadrature stress,P_Q
12	quadrature strain,X_Q

    These values should be 0.000±0.005 volts.

8. If any channel shows values out of the specified range, check the zero offset for that channel on the front of the lock-in amplifier. The + or - switch for zero offset must be depressed depending on the sign of the offset to be removed. Offsets are minimized from the amplifier by rotating the zero-offset potentiometer for that channel, searching for a minimum.

9. If the offset cannot be removed with lock-in amplifier controls, it will be necessary to trim the "zero" potentiometers on the Temperature, Phase, Amplitude board in the multiprogrammer. Refer to diagram on page 25 - R20 for in-phase adjustments and R21 for quadrature. You will find these trim pots on the board marked "Z". The trim pot on the upper edge of the board is for the in-phase adjustment, and the one on the lower edge of the board is for the quadrature adjustment.

CARD LOCATIONS

MULTIPROGRAMMER

Card	Location
Std. Input	100
Remote/Local	200
Logic & Timing	300
Relay	402B
Voltage Monitor	403C
Amplitude/Phase/Temp.	405E
Tension	407G
Heater	409I
Motor	411K
Range	414N
Unit Select	500
Voltage Reg.	600

accepting inputs of parameters from
the operator

Routine for selecting tape file no.
for recording data and accepting
test parameters.

```
0: dsp "RHEOVIBR
ON DATA ACQUISI
TION"
1: gsb "PARAMETE
RS"
2: gsb "COOLDOWN
"
3: gsb "GO"
4: "END":dsp
"Not a null
file - restart"
;end
5: "PARAMETERS":
dim R[20]
6: ent "File #
for this test
?",r24
7: trk 0;fdf
r24;idf r32,
r33;if r33#0;
gto "END"
8: mrk 1,160
9: ent "Sample
#",r31;r31→R[1]
10: ent "Day",
r33;r33→R[2]
11: ent "Month",
r32;r32→R[3]
12: ent "Year",
r34;r34→R[4]
13: ent "Sample
length",r35;
r35→R[5]
14: ent "Sample
width",r36;r36→
R[6]
15: ent "Sample
thickness",r37;
r37→R[7]
16: ent "Max.
temp.",r16;r16→
R[8]
17: ent "Min.
temp.",r17;r17→
R[9]
18: 3.797e3→r10;
r10→R[10]
19: 6.53e-4→r11;
r11→R[11]
20: ent "Frequen
cy",r38;r38→R[1
2]
21: ent "C1",
r41;r41→R[13]
22: ent "C2",
r42;r42→R[14]
23: ent "C3",
r43;r43→R[15]
24: ent "Max.loa
d",r18;r18→R[16
]
```

```
25: ent "Max.
    sample length",
    r15;r15→R[17]
26: ent "Min.
    stress amplitud
    e",r14;r14→R[18
    ]
27: fdf r24;rcf
    r24,R[*]
28: fmt c5,f9.0
29: wrt 16,"smp#
    ",r31
30: spc
31: fmt c3,2x,
    f2.0,c,f2.0,c,
    f4.0
32: wrt 16,"dte"
    ,r32,"/",r33,"/
    ",r34
33: spc
34: fmt c4,e9.3,
    c3
35: wrt 16,"lgh=
    ",r35,"cm"
36: wrt 16,"wdt=
    ",r36,"cm"
37: wrt 16,"thc=
    ",r37,"cm"
38: fmt c3,e9.2,
    c4
39: wrt 16,"+
    T=",r16,"C"
40: wrt 16,"-
    T=",r17,"C"
41: fmt c4,e9.3,
    c3
42: wrt 16,"caP=
    ",r10,"d/V"
43: wrt 16,"caX=
    ",r11,"c/V"
44: wrt 16,"fra=
    ",r38,"Hz"
45: wrt 16,"C1="
    ,r41
46: wrt 16,"C2="
    ,r42
47: wrt 16,"C3="
    ,r43
48: wrt 16,"mxP=
    ",r18,"g"
49: wrt 16,"mxL=
    ",r15,"cm"
50: wrt 16,"MIP=
    ",r14,"g"
51: spc 3
52: dsp "READY
    FOR COOLDOWN???
    ???"
53: stp
54: ret
```

These lines establish printer tape
formats for printing the parameters
entered in earlier steps.

```
55: "COOLDOWN":
56: 1e10→r8
57: gsb "PC4"
58: gsb "RT"
59: if r17>r5;
    jmp 3
60: gsb "STP"
61: jmp -5
62: dsp "***Mini
    mum temperature
    ***"
63: stp
64: ret
65: "GO":
66: flt 3
67: "ALP":-200→r
    17;1→W
68: gsb "RNG"
69: moct;wtb 9,
    170160,20001
70: dsp "I and
    Q stress readin
    g"
71: wait 15000
72: gsb "PC4"
73: wait 15000
74: moct;wtb 9,
    170160,20005
75: .005→D
76: gsb "R"
77: A→r27
78: moct;wtb 9,
    170160,20011
79: .005→D
80: gsb "R"
81: A→r26
82: 2→W
83: moct;wtb 9,
    170040,160001
84: moct;wtb 9,
    170160,20006
85: dsp "I and
    Q strain readin
    g"
86: wait 30000
87: .005→D
88: gsb "R"
89: A→r22
90: moct;wtb 9,
    170160,20012
91: .005→D
92: gsb "R"
93: A→r21
94: r35+1.25e-4*
    r25→r3
95: (F+E)/2→r4
96: gsb "PC4"
97: gsb "RT"
98: gsb "TCO"
99: gsb "IQDE"
100: gsb "PRT"
101: gsb "STP"
102: gsb "RCF"
103: gto "ALP"
104: ret
```

<div style="float:right">

Comments

Routine for controlling sample
tension and measuring temperature
during cooldown in preparation
for a test run.

The main routine for collecting
data

Reading the load transducer

Reading the displacement transducer

94: Correcting sample length to be used
     in the modulus calculation
96: Correcting sample tension
97: Reading temperature
98: Adjusting power to heaters
99: Calculating E and tan δ
100: Printing values of Temperature, E and tan δ
101: Checking for limits
102: Recording data

</div>

```
105: "RT":
106: dsp "Temper
ature reading"
107: 0+N+B
108: mocti;wtb 9,
170160,20020+W
109: -.155+D;
gsb "R"
110: N+1+N;A+
B+B;B/N+r5
111: if N<100;
jmp -2
112: ret
113: "R":
114: 0+A
115: mocti;wtb 9,
170260,30000,
170240
116: wti 0,11;
wti 4,30000;
rdi 4+A;mdec
117: otdA+A;if
A>2047;A-4096+A
118: D*A+A
119: ret
120: "RCF":
121: r24+1+r24
122: trk 0;fdf
r24;mrk 1,56
123: rcf r24,r1,
r7
124: ret
125: "PM":
126: dsp "Tensio
n reading"
127: 0+N;100+E;
.005+D;0+F
128: mocti;wtb 9,
170160,20040+W
129: gsb "R"
130: if A<E;A+E
131: if A>F;A+F
132: N+1+N;if
N<160;jmp -3
133: ret
134: "RNG":
135: gsb "PM"
136: F-E+G;G/
2+G;.707G+G
137: if G>.9;
25+r20;160010+C
;jmp 4
138: if G>.23;
10+r20;160004+C
;jmp 3
139: if G>.09;
2.5+r20;160002+
C;jmp 2
140: if C<.09;
1+r20;160001+C
141: mocti;wtb 9,
170040,C
142: ret
```

Routine for measuring temperature

Routine for reading the multiprogrammer

Routine for recording data

Measuring minimum and maximum of
load sine wave for establishing
tension limits

Routine for autoranging the
lock-in amplifier

```
143: "PRT":
144: flt 3
145: prt "T=";r5
146: prt "E",r8
147: prt "tan",
r9
148: spc 3
149: ret
150: "STP":if
r15<r3;prt "max
.L limit";jmp 5
151: if r16<r5;
prt "max.temp.l
imit";jmp 4
152: if r17>r5;
prt "min.temp.l
imit";jmp 3
153: if r4>r18;
prt "max.load
limit";jmp 2
154: jmp 5
155: moct;wtb 9,
170000,27777
156: fmt 7x,c13
157: wrt 0,"***
LIMITS***"
158: stp
159: ret
160: "+S":
161: moct;wtb 9,
170160,20201,
21001
162: wtb 9,17004
0,20000+W
163: r25+1+r25
164: ret
165: "-S":
166: moct;wtb 9,
170160,20200+W,
20400+W
167: wtb 9,17004
0,20000+W
168: r25-1+r25
169: ret
```

Routine for printing T, E, and tan δ

Routine for checking limits. Program halts if limits are exceeded.

Routine for stepping the motor to add to sample tension.

Routine for subtracting from sample tension.

```
170: "IQDE":
171: r26/r27→A→r
     6;atn(r6)→r6
172: r21/r22→B→r
     1;atn(r1)→r1
173: atn(A)-atn(
     B)→r19;abs(r19)
     →r19
174: tan(r19)→r9
175: flt 3
176: r(r27↑2+
     r26↑2)→r28
177: r28*r20*
     r10→r7
178: r(r22↑2+
     r21↑2)→r23
179: r23*r11→r2
180: r7/r2*r3↑2→
     A
181: r35*r36*
     r37→B
182: A/B→r8
183: 1.24e-4/r3*
     r8*(r36*r37)→Y;
     2.09e-5*Y→Y
184: Y*r2→Y;.5Y→
     Y
185: flt 3
186: ret
187: "PC4":
188: 0→N
189: "DLP":N+1→N
190: if N>3;gto
     "CLP"
191: r8*2.4+32e-
     12→Y
192: r9*1e-10→r1
     3
193: r13+10Y→r12
194: gsb "FM"
195: if E<r13;
     jmp 3
196: if E>r12;
     jmp 7
197: gto "BLP"
198: (r13-E)/
     Y→B;abs(B)+1→B;
     fxd 0
199: B-1→B
200: gsb "+S"
201: if B>0;jmp
     -2
202: gto "DLP"
203: if Y<1e-2;
     gto "CLP"
204: (E-r12)/
     Y→B;abs(B)+1→B;
     fxd 0
205: B-1→B
206: gsb "-S"
207: if B>0;jmp
     -2
208: "BLP":flt
     3;gto "DLP"
209: "CLP":ret
```

Routine for calculating E
and tan δ

Routine for adjusting sample
tension

```
210: "ZEROS":
211: ent Z
212: moct!wtb 9,
     170160,20000+Z
213: .005→D!gsb
     "R"
214: fxd 3!dsp A
215: gto 2
216: "CHECK1":
217: ent Y
218: moct!wtb 9,
     170040,16000+Y
219: stp
220: moct!wtb 9,
     170160,16000
221: gto 10
222: end
223: "AMPS":
224: ent Z
225: moct!wtb 9,
     170160,20000+Z,
     30000
226: moct!wtb 9,
     170040,160002
227: .005→D!gsb
     "R"
228: fxd 3!dsp A
229: gto 227
230: "TCO":
231: if r5>200;
     moct!wtb 9,1700
     40,110024!jmp
     13
232: if r5>120;
     moct!wtb 9,1700
     40,110004!jmp
     12
233: if r5>60;
     jmp 3
234: if r5>20;
     jmp 5
235: if r5>-30;
     jmp 6
236: K-1→K
237: if K=1;moct
     !wtb 9,170040,
     110000!jmp 2
238: if K=0;moct
     !wtb 9,170040,
     110004!2→K!jmp
     6
239: L-1→L
240: if L=1;moct
     !wtb 9,170040,
     110004!2→L!jmp
     4
241: moct!wtb 9,
     170040,110004
242: wait 10000
243: moct!wtb 9,
     170040,110000
244: ret
*13633
```

Routine for checking system zero

This routine is not used.

Routine for checking zero and calibration

Routine for adjusting power to the heaters in temperature chamber

RHEOVIBRON PROGRAM   r Variables

r1   degrees, strain phase angle

r2   cm, strain amplitude

r3   cm, sample length

r4   dynes, average tension static

r5   °C, temperature

r6   degrees, stress phase angle

r7   dynes, stress amplitude

r8 $dynes/cm^2$, E

r9   tan δ

r10   dynes/volt, load calibration

r11   cm/volt, displacement calibration

r12   dynes, PC Max. load-tension control

r13   dynes, PC Min. load-tension control

r14   dynes, Min. stress amplitude

r15   cm, Max. sample length

r16   °C, Max. temperature

r17   °C, Min. temperature

r18   dynes, Max. load

r19   degrees, δ

r20   ranging scale factor "RNG":

r21   volts, quadrature strain, X_Q

r22   volts, in-phase strain, X_T

r23   volts, strain amplitude, X

r24   recording file register "RCF":

r25   "+S" & "-S" counter

r26 volts, quadrature stress, P_Q

r27 volts, in-phase stress, P_I

r28 volts, stress amplitude, P_V

r29

r30

r31 sample #

r32 month

r33 day

r34 year

r35 cm, sample length (initial)

r36 cm, sample width

r37 cm, sample thickness

r38 Hz, frequency

r39

r40

r41 C1

r42 C2

r43 C3